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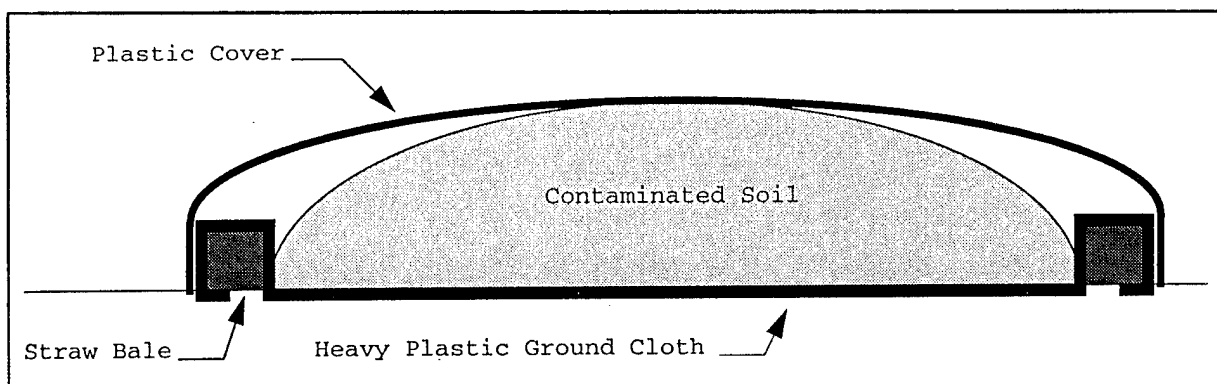
Construction Engineering  
Research Laboratories

USACERL Technical Report 96/47  
February 1996

# Onsite Treatment of Petroleum, Oil, and Lubricant (POL)-Contaminated Soils at Illinois Corps of Engineers Lake Sites

by

Diane K. Mann, Tina M. Hurt, Eva Malkos, Jerry Sims, Scott Twait, and Genie Wachter



To achieve compliance with Federal and state underground storage tank (UST) regulations, the U.S. Army Engineer District, St. Louis decided to remove USTs at Illinois lake sites. The decision was to remove, rather than upgrade, all Corps-owned USTs in the St. Louis District and replace them, where needed, with above-ground storage tanks. During the removal process, leaking USTs were identified at the Illinois lakes of Shelbyville, Carlyle, and Rend. In June 1993, soil borings and samples were taken at each tank location. Laboratory results indicated soil contamination at the

lake sites was greater than Illinois regulatory limits. All USTs were removed in May and June 1994.

This report documents the design and testing of methods to treat soil contaminated by POLs onsite by enhancing aerobic decomposition. The St. Louis District and USACERL developed an inexpensive, easily accomplished method for treating occasional instances of POL-contaminated soil. The method may be of interest to other Corps Districts and military installations.

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Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave Blank)		2. REPORT DATE February 1996		3. REPORT TYPE AND DATES COVERED Final	
4. TITLE AND SUBTITLE Onsite Treatment of Petroleum, Oil, and Lubricant (POL)-Contaminated Soils at Illinois Corps of Engineers Lake Sites				5. FUNDING NUMBERS MIPR CELMS-ED 95-108	
6. AUTHOR(S) Diane K. Mann, Tina M. Hurt, Eva Malkos, Jerry Sims, Scott Twait, and Genie Wachter					
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Construction Engineering Research Laboratories (USACERL) P.O. Box 9005 Champaign, IL 61826-9005				8. PERFORMING ORGANIZATION REPORT NUMBER  TR 96/47	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Engineer District, St. Louis ATTN: CELMS 210 Tucker Blvd. North St. Louis, MO 63101-1986				10. SPONSORING / MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES Copies are available from the National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161.					
12a. DISTRIBUTION / AVAILABILITY STATEMENT  Approved for public release; distribution is unlimited.				12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words)  To achieve compliance with Federal and state underground storage tank (UST) regulations, the U.S. Army Engineer District, St. Louis decided to remove USTs at Illinois lake sites. The decision was to remove, rather than upgrade, all Corps-owned USTs in the St. Louis District and replace them, where needed, with aboveground storage tanks. During the removal process, leaking USTs were identified at the Illinois lakes of Shelbyville, Carlyle, and Rend. In June 1993, soil borings and samples were taken at each tank location. Laboratory results indicated soil contamination at the lake sites was greater than Illinois regulatory limits. All USTs were removed in May and June 1994.  This report documents the design and testing of methods to treat soil contaminated by POLs onsite by enhancing aerobic decomposition. The St. Louis District and USACERL developed an inexpensive, easily accomplished method for treating occasional instances of POL-contaminated soil. The method may be of interest to other Corps Districts and military installations.					
14. SUBJECT TERMS environmental compliance underground storage tanks contaminated soils  Petroleum, Oil, and Lubricant (POL)				15. NUMBER OF PAGES 68	
				16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified		20. LIMITATION OF ABSTRACT SAR	

## Foreword

This study was conducted for the U.S. Army Engineer District St. Louis under Military Interdepartmental Purchase Request (MIPR) No. CELMS-ED 95-108, dated 28 October 1994. The St. Louis District technical monitor was Genie Wachter.

The research was performed by the Environmental Processes Division (PL-N) of the Planning and Management Laboratory (PL), U.S. Army Construction Engineering Research Laboratories (USACERL). The USACERL principal investigator was Dr. Diane K. Mann (PL-N). Associate investigators were Tina M. Hurt, Scott Twait, and Eva Malkos (PL-N). Jerome L. Benson is Acting Chief, CECER-PL-N; L. Michael Golish is Operations Chief, CECER-PL; and Dr. David M. Joncich is Chief, CECER-PL. The USACERL technical editor was Linda L. Wheatley, Technical Resources Center.

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# Contents

<b>SF 298</b>	1
<b>Foreword</b>	2
<b>List of Tables and Figures</b>	5
<b>1 Introduction</b>	7
Background	7
Scope	8
Objective	9
Approach	9
Metric Conversion Factors	9
<b>2 History of Tank Sites</b>	10
Lake Shelbyville	10
Rend Lake	11
Carlyle Lake	13
<b>3 Treatment Designs and Execution</b>	14
Basic Treatment Design	14
Site Design	16
<b>4 Field Monitoring and Maintenance</b>	24
Monitoring Procedures	24
Windrow Envelopes	24
Treatment and Stock Piles	26
<b>5 Laboratory Procedures</b>	30
Soil Samples	30
Performance of Analyses	30
Determination of Concentrations	31
Graphing and Analysis Results	31
<b>6 Conclusions</b>	40
<b>7 Lessons Learned</b>	41
Laboratory	41
Field	43

<b>References</b> .....	44
<b>Appendix A:</b> Lake Site Remediation Patterns .....	45
<b>Appendix B:</b> Statistical Analysis System Program Output .....	56
<b>Distribution</b>	



## List of Tables and Figures

### Tables

1	POL contaminants present in samples of June 1993 site assessment .....	11
2	Properties of Lake Shelbyville soil samples .....	12
3	Nutrient analysis of soil samples collected at Carlyle Lake on 19 May 1994 .....	13
4	Monitoring and maintenance record for Lake Shelbyville .....	25
5	Monitoring and maintenance record for Carlyle Lake .....	27
6	Monitoring and maintenance record for Rend Lake .....	28
7	Gas chromatograph setup and Tekmar LSC 2000 setup for Method 1 .....	32
8	Example regression analysis performed on standards of toluene .....	33
9	Temperature readings (°C) at Lake Shelbyville .....	36
10	Temperature readings (°C) at Rend Lake .....	37
11	Temperature readings (°C) at Carlyle Lake .....	38
12	Laboratory equipment .....	42
13	Field equipment .....	42

### Figures

1	Cross section of windrow envelope construction showing (a) contaminated soil placed on ground cloth about 2 ft from long edge and (b) ground cloth folded around soil to create envelope .....	15
2	Cross section of a treatment pile .....	17
3	Layout of Lake Shelbyville maintenance yard treatment site (16 May 1994) .....	18

4	Layout of Lake Shelbyville Boneyard treatment site (25 May 1994) .....	20
5	Layout of Carlyle Lake treatment site (14 June 1994) .....	21
6	Layout of Rend Lake treatment site (15 June 1994) .....	22
7	Layout of Rend Lake site for final treatment stage (22 November 1994) .....	23
8	Concentration of toluene versus GC output area .....	33
9	Plot of soil temperature for black and white covered soil relative to air temperatures .....	39
A1	Remediation pattern of Windrow 1 (WR1), Lake Shelbyville .....	47
A2	Remediation pattern of WR2, Lake Shelbyville .....	47
A3	Remediation pattern of WR3, Lake Shelbyville .....	48
A4	Remediation pattern of WR4, Lake Shelbyville .....	48
A5	Remediation pattern of Treatment Pile 1 (TP1), Lake Shelbyville .....	49
A6	Remediation pattern of TP2, Lake Shelbyville .....	49
A7	Remediation pattern of WR1, Carlyle Lake .....	51
A8	Remediation pattern of WR2, Carlyle Lake .....	51
A9	Remediation pattern of WR3, Carlyle Lake .....	52
A10	Remediation pattern of treatment pile, Carlyle Lake .....	52
A11	Remediation pattern of WR1, Rend Lake .....	54
A12	Remediation pattern of WR2, Rend Lake .....	54
A13	Remediation pattern of WR3, Rend Lake .....	55
A14	Remediation pattern of TP1, Rend Lake .....	55

# 1 Introduction

## Background

Soil contaminated with petroleum, oil, and lubricant (POL) is often a problem at U.S. Army sites with underground storage tanks (USTs) of questionable integrity or spills during operations and training. During the process of removing all Corps-owned USTs in the U.S. Army Engineer District St. Louis, leaking USTs were identified at Illinois lakes of Shelbyville, Carlyle, and Rend. Soil samples taken at each tank location indicated soil contamination at the lake sites was greater than Illinois regulatory limits. These sites were selected for an experiment to treat POL-contaminated soil undertaken jointly by the St. Louis District and the U.S. Army Construction Engineering Research Laboratories (USACERL).

Research for this report focused on enhancing a treatment process for POL-contaminated soils based on aerobic decomposition of organic waste. Physical, chemical, and biological capabilities of soil can be manipulated to improve efficiency of biodegradation. Aerobic bacteria and fungi play the major role in biodegrading petroleum wastes (Atlas 1984). Offensive odors can be avoided because noxious products such as hydrogen sulfide, amines, and mercaptans are not produced in soils with an oxygenated environment. In contrast, soil environments undergoing anaerobic degradation are slower, incomplete, and favor leaching (Casarini et al. 1990).

Ideal soil conditions for aerobic biodegradation by indigenous microorganisms depend on factors such as aeration, pH, temperature, moisture, and nutrients. Research has established that bacteria are especially adept at mediating biodegradation of compounds common to petroleum fuels (Autry and Ellis 1992) and several parameters have been tested. Optimum pH for hydrocarbon biodegradation in soil lies between 6.5 and 7.5 (Dibble and Bartha 1979). Temperatures between 20 and 30 °C maximize POL biodegradation in soil, whereas the process stops at 5 °C (Atlas 1984) or requires special conditions (Huddleston and Cresswell 1976). Biodegradation of simple and complex organic material in soil is greatest commonly at 50 to 70 percent of soil water-holding capacity (Pramer and Bartha 1972). Continuing research on fertilization suggests that addition of nitrogen, phosphorus, and potassium may be beneficial to speed up the biodegradation process; amounts to be added vary with type of soil and nutrients occurring naturally.

### ***Lake Shelbyville***

Lake Shelbyville extends north and east from the community of Shelbyville. The dam that forms the lake stands 110 ft above the bed of the Kaskaskia River and creates a lake covering 11,000 acres surrounded by 172 mi of forested shoreline. Constructed in the 1960s, the lake offers many recreational opportunities and serves as flood and wildlife protection. In preparation for removing a UST in the maintenance yard near the Lake Shelbyville Visitor Center located in the Dam East Recreation Area, testing showed that a gasoline tank had leaked.

### ***Carlyle Lake***

Carlyle Lake extends north and east from the community of Carlyle. Also on the Kaskaskia River and constructed in the 1960s, the lake covers 26,000 acres with 11,000 acres of public land. Many recreational opportunities are available in addition to flood protection and conservation. At the Dam West Recreation Area near the administration building, an underground heating oil tank was found to have leaked, as was an underground diesel tank in the maintenance yard, when testing was done before removal.

### ***Rend Lake***

Rend Lake extends north and slightly west of the community of Benton. Another 1960s project, the dam impounds the main branch of the Big Muddy River and its tributaries. Surrounding the 19,000 acres of water is 21,000 acres of public land that provides recreation, conservation, and flood control. The UST leaking at this site was a diesel tank in the maintenance yard near the U.S. Army Corps of Engineers Administration Office.

## **Scope**

Although temperature, fertilization, and moisture were elements of the research at the lake sites, the research design emphasized aeration. Controlled experiments using treatment piles versus windrow envelopes and fertilized windrow envelopes versus unfertilized windrow envelopes were studied for the rate of contaminant removal by indigenous microorganisms present in the contaminated soil. Soil was considered "clean" when State of Illinois Cleanup Objectives (Illinois Environmental Protection Agency 1993) were reached or contaminants were no longer detected. Many studies have indicated that encouraging microbial decomposition of POL contaminants results in fertile, useable soil and reduces monitoring, maintenance, and cost of landfilling.

The St. Louis District joined USACERL to develop an inexpensive, easily accomplished method for treating occasional instances of POL-contaminated soil. This report documents the method and may be of interest to Army installations and other Corps Districts.

## Objective

The objective of this research was to design and test methods to treat POL-contaminated soil onsite by enhancing aerobic decomposition.

## Approach

Chapter 2 contains a history of the sites involved in the research project. Chapter 3 is the general design of the research project, and Chapter 4 presents monitoring specifics at each location. Chapter 5 gives laboratory procedures and analysis results with detailed information shown in Appendices A and B. All POL-contaminated soils at two sites and most at the third site were successfully treated over summer months (Chapter 6) and reused at project sites with conclusive research results. Chapter 7 discusses lessons learned during this project and suggests other appropriate applications.

## Metric Conversion Factors

U.S. standard units of measure are used throughout this report. A table of metric conversion factors is presented below.

1 in.	=	25.4 mm
1 sq ft	=	0.093 m <sup>2</sup>
1 mi	=	1.61 km
1 lb	=	0.453 kg
1 gal	=	3.78 L
°F	=	(°C × 1.8) + 32
1 cu yd	=	0.765 m <sup>3</sup>

## 2 History of Tank Sites

To achieve compliance with Federal and state UST regulations, the St. Louis District decided to remove USTs at Illinois lake sites. The decision was to remove (rather than upgrade) all Corps-owned USTs in the St. Louis District and replace them, where needed, with aboveground storage tanks. During the removal process, leaking USTs were identified at the Illinois lakes of Shelbyville, Carlyle, and Rend.

In June 1993, soil borings and samples were taken at each tank location during a site assessment. Laboratory results indicating soil contamination at the lake sites were greater than Illinois regulatory limits allow (Table 1). All USTs were removed in May and June 1994.

### Lake Shelbyville

Steel 1000-gal diesel and 2000-gal gasoline USTs at the maintenance area were removed in May 1994. The two tanks were in use until removed. The diesel UST was installed in 1970; the gasoline UST was installed in 1982 to replace a leaking 1000-gal UST installed in 1970. Maintenance personnel detected the leak by observing water in the gasoline rather than by a noticeable change in fuel consumption. They observed small pinholes in the UST when it was removed in 1982. Contaminated soil was left in the pit when the new UST was installed; therefore, when the two USTs were removed in 1994, workers anticipated finding a large quantity of previously contaminated soil.

As expected, gasoline contaminated soil was excavated above and below the USTs. All contaminated soil samples from this site were coarse in texture and deficient in carbon and nutrients (Table 2). When the USTs were pulled from the ground and inspected, they were in excellent condition with no leaks in the piping system. Therefore, all contamination was attributed to the UST removed in 1982.

Table 1. POL contaminants present in samples of June 1993 site assessment.

Lake Shelbyville (Gasoline Tank)		
Constituent	Contamination Level ppm	Cleanup Goal ppm
Benzene	7.3	0.005
BTEX	180	11.705
Carlyle Lake (Heating Oil Tank)		
Constituent	Contamination Level ppm	Cleanup Goal ppm
Benzene	0.8	0.005
Napthalene	6.9	0.025
Rend Lake (Diesel Fuel Tank)		
Constituent	Contamination Level ppm	Cleanup Goal ppm
Benzene	0.75	0.005
Napthalene	4.3	0.025

## Rend Lake

Two 1000-gal steel USTs, one for diesel and one for gasoline, were installed in 1970 in the maintenance area. In 1987, a 6000-gal steel UST was installed for gasoline. After the 1987 installation, two 1000-gal steel USTs were connected and used for diesel fuel storage. All three tanks were in use until April 1994 and were removed in May 1994.

The USTs were in good condition with little rust and no apparent holes when they were removed and inspected. Consequently, contamination is thought to have occurred when the two 1000-gal USTs were connected and from overfills of diesel fuel.

Table 2. Properties of Lake Shelbyville soil samples.

Soil	Organic Carbon (%)	P (mg/kg)	K (mg/kg)	Mg (mg/kg)	Ca (mg/kg)	pH	Texture			
							CEC (meq/100g)	Sand (%)	Silt (%)	Clay (%)
WR	0.35	2	57	280	3200	8.1	18.5	62	19	19
TP-S	0.12	1	21	130	4100	8.2	21.6	86	7	7
TP-C	0.35	2	62	325	3400	8.1	19.9	54	23	23
UND	0.87	2	66	190	2150	7.8	12.5	14	55	31

WR = windrow

TP-S = sandy treatment pile

TP-C = clayey treatment pile

UND = undisturbed, uncontaminated soil

CEC = cation exchange capacity



## Carlyle Lake

Two 1000-gal steel USTs for gasoline and diesel fuel were installed in 1966 in the maintenance area. When water was discovered in the gasoline UST in 1985, its use was discontinued. The diesel UST was used until its removal in May 1994. In addition, a 560-gal steel UST for heating oil adjacent to the administration building was removed. It was installed in 1966 and abandoned in 1982 when the building was converted to natural gas heat.

Laboratory results from the abandoned gasoline UST did not indicate contamination. Fluid in the diesel UST measured 8.5 in. total (7.5 in. of water and 1 in. of fuel). A soil boring from the heating oil UST site indicated soil contamination above Illinois regulatory limits (Table 1). Eleven in. of water and 21 in. of fuel were left in this UST.

When the USTs were removed, contaminated soil was found at both the maintenance area and administration building. Small nutrient levels existed in soil samples taken from the sites (Table 3). The tanks were rusted and pitted with holes, and contamination was due to fuel remaining in them.

**Table 3. Nutrient analysis of soil samples collected at Carlyle Lake on 19 May 1994.**

Parameter	Treatment Pile	Stock Pile	Method
Potassium (%)	625	621	6010
TKN (%)	305	316	351.2
Total Phosphate (%)	149	137	365.2
Total Solids (%)	90.1	87	160.3

### 3 Treatment Designs and Execution

#### Basic Treatment Design

POL-contaminated soil was estimated at 50 cu yd each for the three lake locations. Researchers visited each site and examined suggested treatment areas. If more than one treatment area was available, the area selected had the best road access, was near to a water source, and had an adequately flat, smooth surface for nylon-reinforced plastic ground cloths.

Basic research design was to compare dissipation of POLs in contaminated soil under varying conditions. The main emphasis was on comparing soil cleanup rates of treatment piles versus specially designed windrow envelopes. Black or white covers and windrow envelopes were used to compare temperatures between the black and white surfaces. Aeration, fertilization, and watering were performed on selected windrows and compared to control windrows.

#### *Windrow Envelopes*

Black windrow envelopes, with one exception, were created from 45 by 12-ft sheets of 20-mil-reinforced plastic. All envelopes with a white surface exposed to the sun and one black envelope were of the same dimensions, but of 9-mil-reinforced plastic sheets instead of 20-mil-reinforced plastic sheets. Each windrow envelope was created by placing soil on the plastic sheet 2 ft from one of the long edges and 2.5 ft from the short edges (Figure 1). Each elongated pile of soil measured approximately 4-ft wide and 2-ft high. Because the elongated mound of dirt tapered to a ridge, total soil was approximately 5 cu yd per windrow envelope. The extra 2.5 ft of plastic at each end of the windrow and the 2 ft of liner along one side of the elongated mound of soil and 6 ft along the opposing side were folded over and weighted down. The created envelope encased the contaminated soil to prevent rain from entering and leachate from escaping.

#### *Treatment Piles*

The size of treatment piles varied from site to site. Rectangular ground cloths of 20-mil-reinforced plastic were used. Five-ft sections on each side of the rectangular sheet were kept clear of soil, and this portion of sheet was folded over bales of straw to create

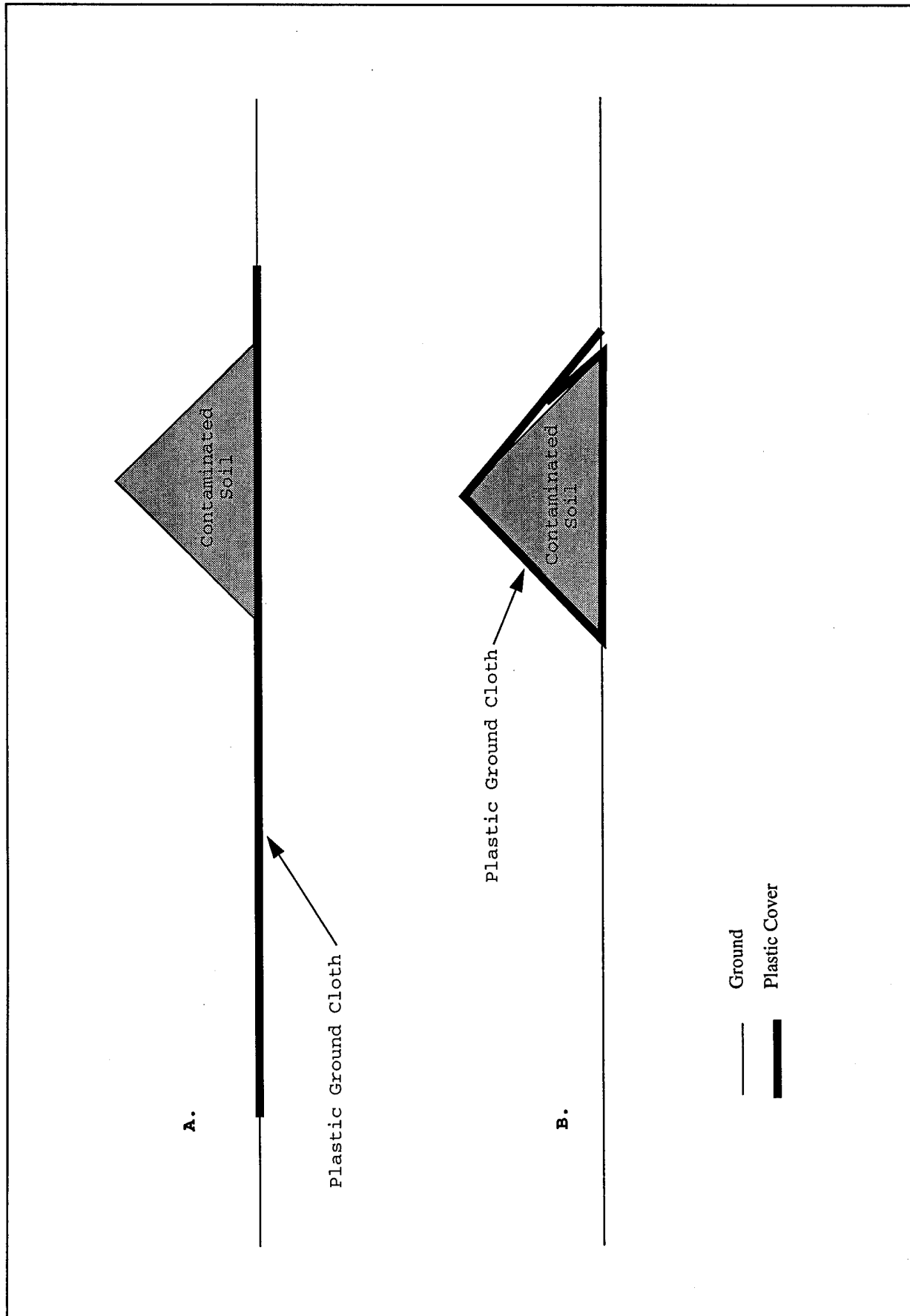


Figure 1. Cross section of windrow envelope construction showing (a) contaminated soil placed on ground cloth about 2 ft from long edge and (b) ground cloth folded around soil to create envelope.

a berm around each treatment pile (Figure 2). Rectangular covers of 9-mil-reinforced plastic measuring 10 ft greater than the rectangular ground cloths were placed over treatment piles and weighted down with sandbags.

### ***Stock Piles***

Stock piles were not part of the original design, but a result of underestimating contaminated soil at the sites. The contractor placed unplanned quantities of contaminated soil on a large clear plastic sheet and covered the soil with another clear plastic sheet. Clear, unreinforced plastic held up poorly, so soil was moved as soon as possible to windrow envelopes for treatment.

## **Site Design**

### ***Lake Shelbyville Site***

Storage tanks to be removed at Lake Shelbyville were under a concrete pad in a maintenance yard. The original plan was to create two windrow envelopes and two treatment piles within the fenced maintenance yard with liners placed on the paved yard surface. Each windrow envelope was designed to hold approximately 5 cu yd of soil and the estimated 40 cu yd of soil remaining were to be placed in treatment piles. Successfully treated soil in envelopes was hauled away to use as fill or ground cover. Soil from treatment piles was windrowed until piles were small enough that POLs had decreased significantly by aerobic decomposition.

Two liners measuring 45 ft by 12 ft of 20-mil-reinforced plastic were oriented east to west and used for windrow envelopes (Figure 3). Both windrow envelopes were black. Each elongated pile of soil was approximately 40-ft long by 4-ft wide by 2-ft high and accommodated approximately 5 cu yd per windrow envelope. Two ground cloths, each 30 ft by 30 ft of 20-mil-reinforced plastic, were to hold two treatment piles of approximately 20 cu yd each (Figures 2 and 3). Nine-mil-reinforced plastic sheets 40 ft by 40 ft were used to cover the treatment piles and were weighted down with sandbags.

Approximately 135 cu yd of POL-contaminated soil was removed at the Lake Shelbyville maintenance area, 85 cu yd more than estimated. The contractor placed the additional stock pile on a large clear plastic sheet covered with clear plastic.

Because of the unexpected quantity of POL-contaminated soil excavated, the Shelbyville plan had to be modified. Because windrow envelopes reached treatment goals more quickly, additional windrow envelopes were created. Contaminated soil from the

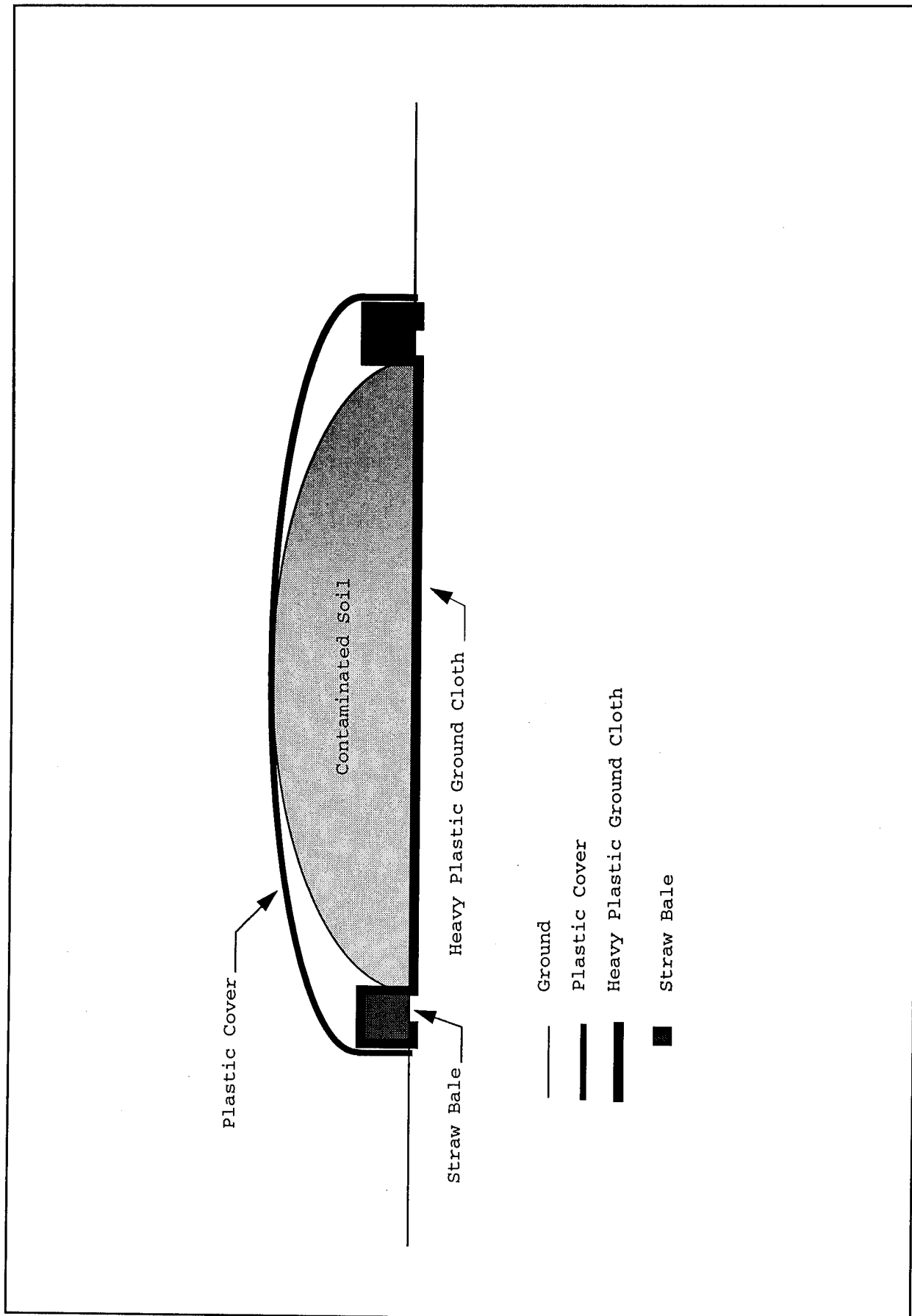


Figure 2. Cross section of a treatment pile.

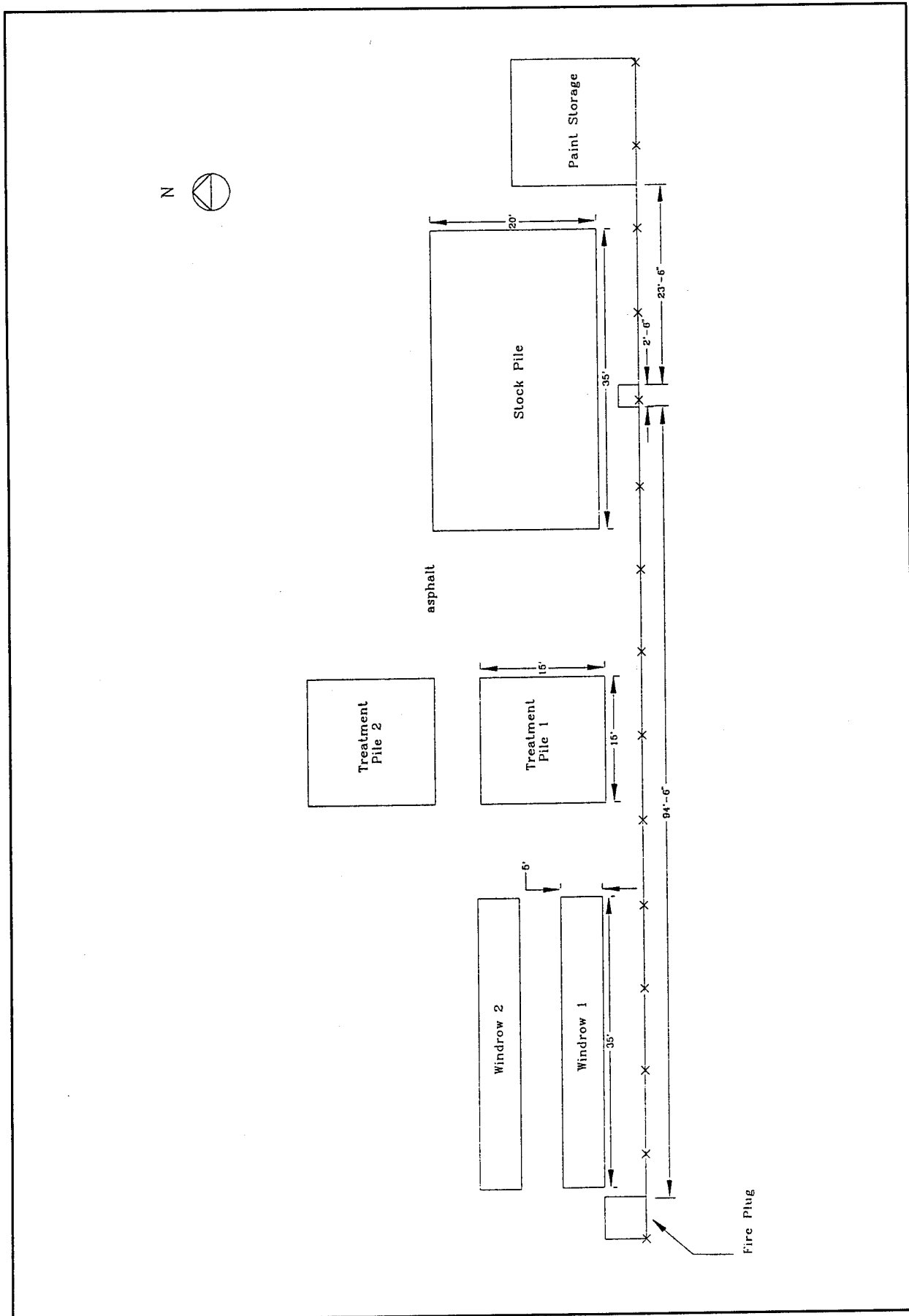


Figure 3. Layout of Lake Shelbyville maintenance yard treatment site (16 May 1994).

stock pile was moved to a nearby site referred to as the "Boneyard" and placed in four windrows and a treatment pile (Figure 4). Windrow envelopes were oriented east to west at the Boneyard. Two treatment piles at the original maintenance site were continued until the acceptable limits were reached.

### ***Carlyle Lake Site***

POL-contaminated soil from leaking USTs was found at two Carlyle Lake locations. Contaminated soil removed from the maintenance yard and administration building on 18 May 1994 was moved approximately 1/4 mile to an area near the radio tower. Based on previous experience from Lake Shelbyville, a stock pile for over 50 cu yd of soil was incorporated into the original design.

The treatment area at Carlyle Lake was a strip of irregular and partially weed- and grass-covered land parallel to the radio tower road. Capitalizing on the smoothest, flattest, rock-free portions, the original design (Figure 5) incorporated two windrow envelopes running north to south, one windrow envelope oriented east to west, an 18 sq ft treatment pile, and a stock pile. All three windrows had black envelopes. On 14 June, contaminated soil from the treatment pile was spread in a fourth windrow running north to south, for which the plastic ground cloth was folded to create a white envelope.

After successful treatment in the four windrow envelopes, soil was removed and used. POL-contaminated soil from the treatment pile and stock pile replaced the treated soil in windrow envelopes, and this process was repeated twice. By then, the stockpiled soil had been depleted and soil remaining in the treatment pile had reached the levels of the treatment goal.

### ***Rend Lake Site***

A sloping, relatively flat area of ground, well covered with grass, was made available for a treatment site at Rend Lake. The site was approximately 50 yd southwest of the maintenance yard where a tank leak followed by numerous instances of diesel overfills had caused POL contamination of soil.

Design for this site included three windrow envelopes, a rectangular treatment pile oriented north to south, and a large stock pile. Soil taken from the treatment pile was used for a fourth windrow envelope 3 weeks after the site was established (Figure 6). After successful treatment of the four windrows, soil from the stock and treatment piles was windrowed, with as many as seven windrow envelopes active at one time (Figure 7). Each time a new windrow was established with untreated soil, it was given a new number.

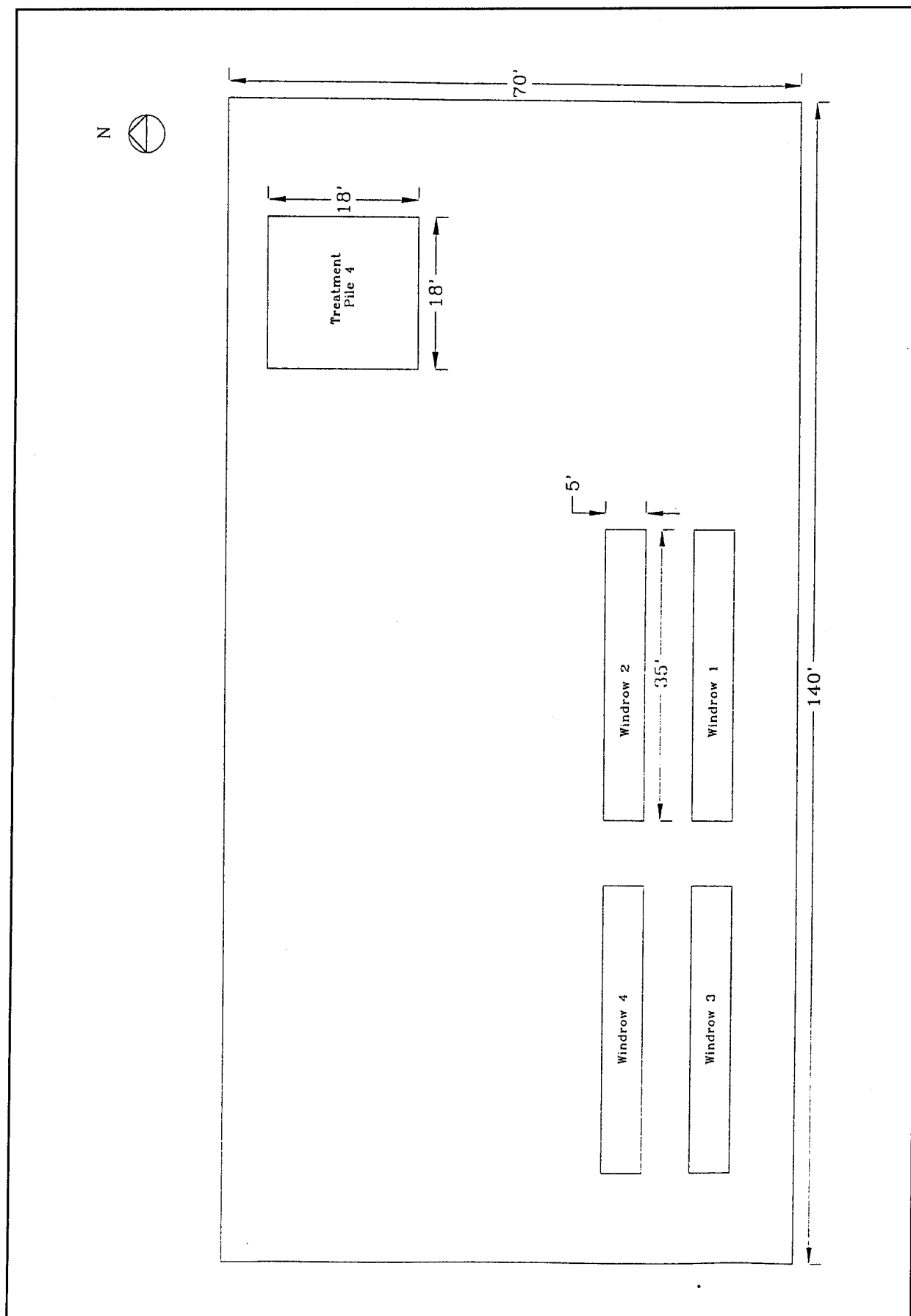


Figure 4. Layout of Lake Shelbyville Boneyard treatment site (25 May 1994).



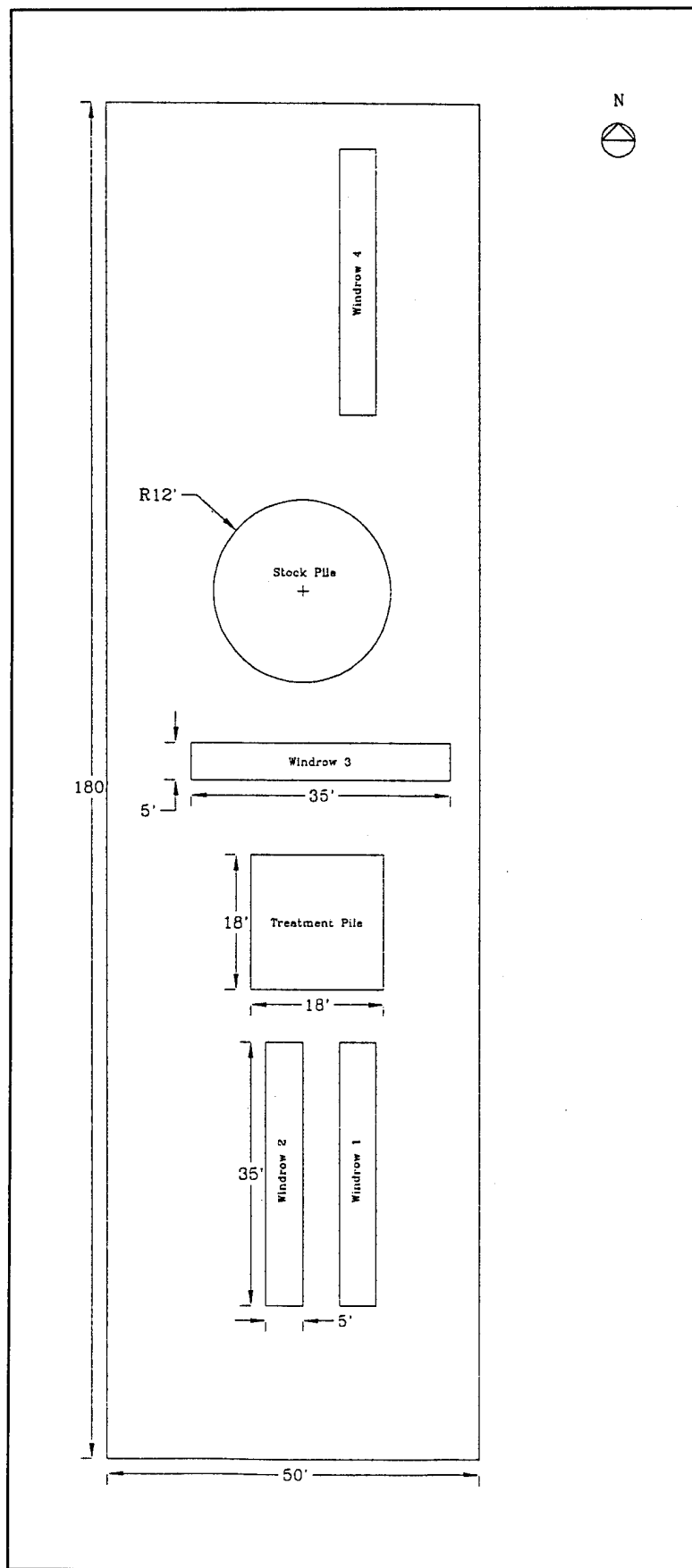


Figure 5. Layout of Carlyle Lake treatment site (14 June 1994).

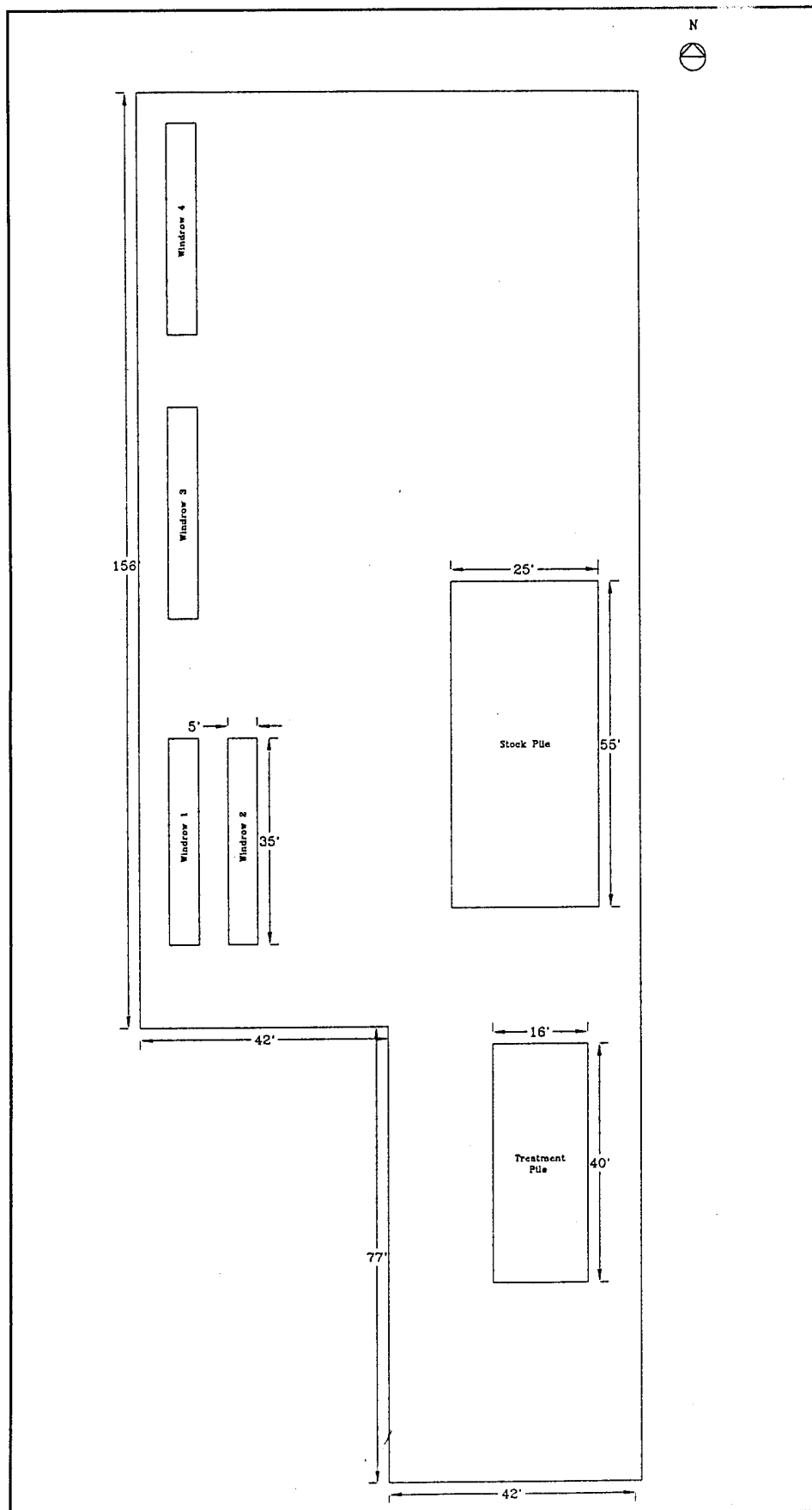


Figure 6. Layout of Rend Lake treatment site (15 June 1994).

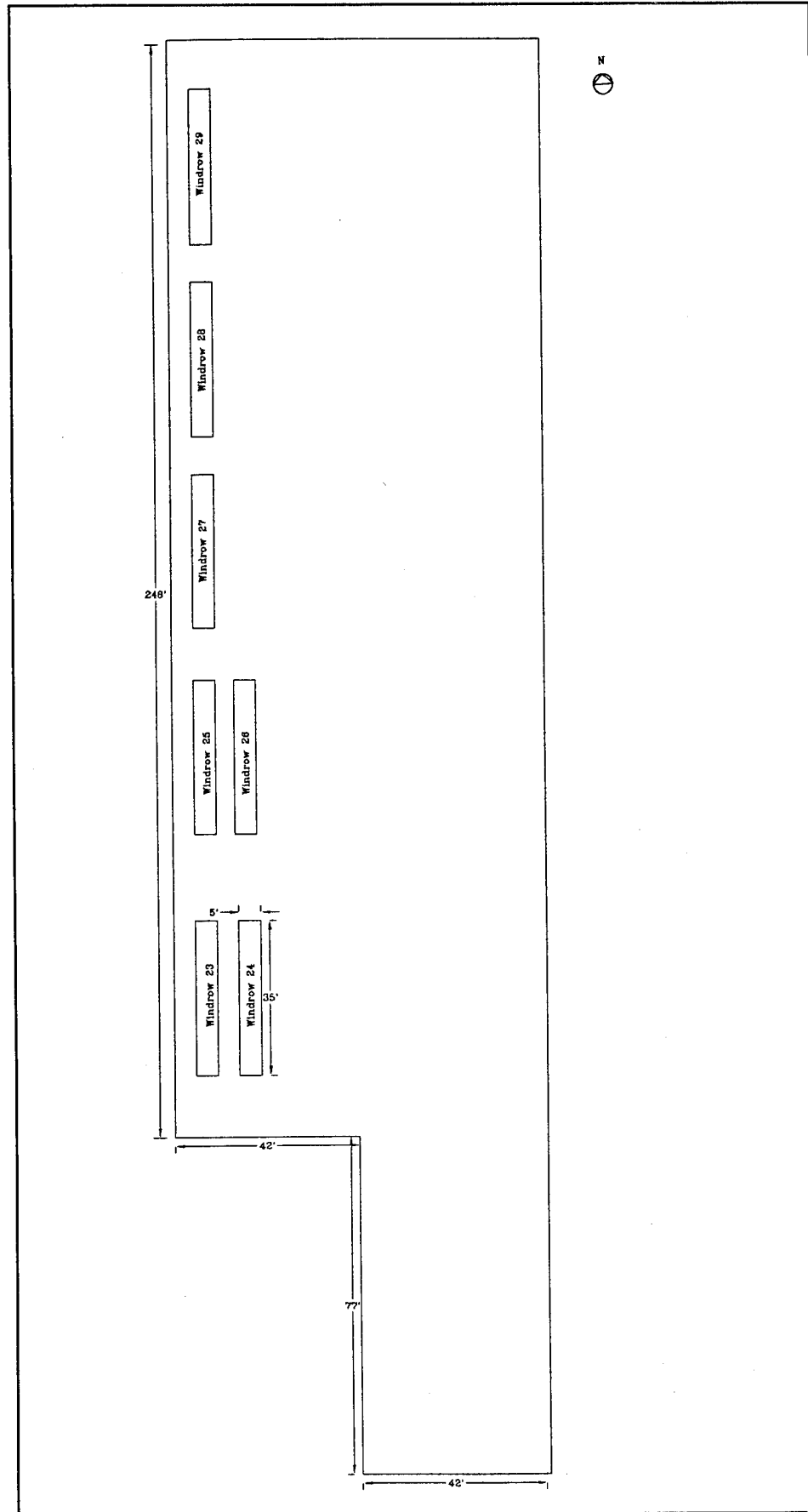


Figure 7. Layout of Rend Lake site for final treatment stage (22 November 1994).

## 4 Field Monitoring and Maintenance

### Monitoring Procedures

Sites were checked weekly. Sampling and temperature data for each windrow envelope and treatment pile were collected. Windrow envelopes also were turned, fertilized, or watered according to research design. Location and depth of all soil samples were recorded on the sample's label and in a field notebook. Soil samples were preserved in airtight sample jars filled to the top to minimize air space and placed in a cooler with ice until they could be placed in refrigeration at the laboratory.

Temperatures from treatment piles and windrow envelopes were recorded to compare temperature differences between soil under black covers with soil under white covers. Surface temperatures of piles and windrow envelopes were recorded for comparison to temperatures recorded at a depth of approximately 6 in. Atmospheric temperatures also were recorded.

### Windrow Envelopes

Windrow envelopes were most susceptible to being shifted by the wind. The first check at Lake Shelbyville required over an hour of repositioning and weighting sections of several windrow envelopes. However, windrow envelope design facilitated soil turning, which was done by hand. For example, windrow envelopes 3 and 4 at Lake Shelbyville were turned the first week (after being established at week 9) and windrow envelope 4 was turned weekly thereafter (Table 4) to compare treatment time with windrow envelopes 1 and 2, which were not turned at all. Sections of a windrow envelope with predominantly clay soil were the most difficult to turn because of heavy clods, at times as large as a cubic foot. First turning of a windrow envelope took an average of 2-1/2 h. Repeated turnings tended to break up the soil into smaller clods, which made the turning easier and faster. Under ideal conditions, a windrow envelope was turned in less than 2 h. Turning time included opening the envelope, shifting the soil row approximately 3 ft, refolding the envelope, and weighting it closed with sandbags.

Table 4. Monitoring and maintenance record for Lake Shelbyville.

Pile	Week																	
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
Windrow 1	S		S	S	S	S, W	S, W	S, W	S	R								
Windrow 2	S		S	S	S	S, W	S, W	S, W	S	R								
Windrow 3									S	S	T, F, S	T, S, W	T, S	S		ready to move		R
Windrow 4									S	S	T, F, S	S, W	S	S		ready to move	S	R
Windrow 5																		S
Windrow 6																		S
Treatment Pile 1			S		S	S	S	S	S	S	S	S	S	S			S	S
Treatment Pile 2/3	S		S		S	S	S	S	S	S	S	S	S	S			S	S

Pile	Week																		
	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Windrow 5	S	S	S	S	S	S	S		S		S	R							
Windrow 6	S	S	S	S	S	S	S		S		S	R							
Windrow 7										S	S	S							
Windrow 8										S	S	S							
Windrow 9										S	S	S							
Windrow 10										S	S	S							
Treatment Pile 1		S	S	S	S	S, T	S		S		S	S, T, F							
Treatment Pile 2/3		S	S	S	S	S	S		S		S	S, T, F							
Treatment Pile 4										S	S	S							

S = sampled  
 T = turned  
 F = fertilized  
 R = removed  
 W = watered

Windrow envelope fertilization required mixing in approximately 1 lb of 28:3:6 lawn fertilizer per windrow. Fertilizer was sprinkled the length of the windrow from a measuring cup and mixed into the soil with a shovel. This process usually took 15 min per windrow envelope in addition to time spent turning the envelope.

Weather conditions and retention of moisture in the envelopes minimized the need for watering (Table 5). Watering was by hose and typically took 15 min per envelope.

### Treatment and Stock Piles

Treatment piles were turned with a backhoe at Lake Shelbyville and Carlyle Lake (Tables 4 and 5). Treatment piles were not watered (Tables 4, 5, and 6), but treatment piles at Lake Shelbyville were fertilized and turned before being covered for the winter.

Stock piles were not fertilized, mixed, or watered. Turning, fertilization, and watering of contaminated soil were started after soil was removed from the pile and placed in a windrow envelope.

Table 4. (Cont'd).

	Week								
	37	38	39	40	41	42	43	44	45
Windrow 7								S	R
Windrow 8								S	R
Windrow 9								S	R
Windrow 10								S	R
Treatment Pile 1								S	R
Treatment Pile 2/3								S	R
Treatment Pile 4								S	R

S = sampled  
R = removed

Table 5. Monitoring and maintenance record for Carlyle Lake.

Pile	Week																						
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22
Windrow 1	S		S	T, S	T, F, S	T, S	T, S	S	T, S, W	T, S	T, S, N	R											
Windrow 2	S		S	S	S	S	S	S	S, W	S	S, N	S	S	T, F, S	S	S	R						
Windrow 3	S		S	S	S	S	S	S	S, W	S	S, N	R											
Windrow 4						S	S	S	S, W	S	S, N	S	S	S	R								
Windrow 5												S	S	S	S	S	R						
Windrow 6												S	T, F, S	S	S	S	R						
Windrow 7															S	T, S	S	S	S	R			
Windrow 8																	S	S	S	R			
Windrow 9																	S	S	S	R			
Windrow 10																	S	S	S	R			
Treatment Pile			S	S	S	S	S	S	S		S, N	spread out	S	S	added SP		S		S	S	S, mixed	S	R

S = sampled  
 T = turned  
 F = fertilized  
 W = watered  
 N = rain  
 R = removed  
 SP = spread out

Table 6. Monitoring and maintenance record for Rend Lake.

Pile	Week																	
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
Windrow 1	S	S	S	S	S	S	S	S	S	S	S	S	S	R				
Windrow 2	S	S	S	S	S	S	S	S	S	S	S	S	S	R				
Windrow 3	S	T, S	T, S	T, S	T, F, S	T, S	T, S	T, S	T, S	T, S	T, S	T, S	T, S	R				
Windrow 4				S	S	S	S	S	S	S	R							
Windrow 5											S	S	S	S	S	Ready to move	R	
Windrow 6														S	T, S	S		S
Windrow 7														S	S	S	S	S
Windrow 8														S	S	S	S	S
Windrow 9																	S	S
Treatment Pile 1	S		S	S	S	S	S	S		S	S	S	S				R	

S = sampled  
 T = turned  
 F = fertilized  
 R = removed



Table 6. (Cont'd)

	Week										
	18	19	20	21	22	23	24	25	26	27	28
Windrow 6	S	R									
Windrow 7	S	R									
Windrow 8	S	R									
Windrow 9	S	R									
Windrow 10		S	S	R							
Windrow 11		S	S	R							
Windrow 12		S	S	R							
Windrow 13		S	S	R							
Windrow 14				S	S	S	S	S	R		
Windrow 15				S	S	S	R				
Windrow 16				S	S	S	R				
Windrow 17				S	S	S	R				
Windrow 18							S	S	R		
Windrow 19							S	S	R		
Windrow 20							S	S	S	S	R, S
Windrow 21							S	S	R		
Windrow 22							S	S	R		
Windrow 23							S	S	R		
Windrow 24									S	S	R, S
Windrow 25									S	S	R, S
Windrow 26									S	S	R, S
Windrow 27									S	S	R, S
Windrow 28									S	S	R, S
Windrow 29									S	S	R, S

S = sampled  
R = removed

## 5 Laboratory Procedures

Laboratory work was based on U.S. Environmental Protection Agency (EPA) Method 5030A, *Purge-and-Trap* (EPA, July 1992) and Method 8260A, *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique* (EPA, November 1992).

### Soil Samples

Contaminated soil samples from windrow envelopes and treatment piles were collected in airtight 4 oz mason jars that were labeled and logged in a journal. Jars were packed tightly, filled to the top, and immediately placed in a cooler. At the laboratory, the samples were placed in a refrigerator or chiller room at approximately 5 °C. Samples were usually analyzed within 2 days of collection; remaining soil was stored for the duration of the project.

Four grams of each soil sample were placed into a 40 mL vial and coded to identify contents. Ten mL of purge-and-trap grade methanol was added to each 40 mL sample vial before the vial was placed in a reciprocating shaker for 2 min at 180 oscillations per minute. After contents had settled (approximately 2 min), the clear liquid was extracted with a disposable pipette, placed in a 1.5 mL vial, clamped with a polytetrafluoro ethylene (PTFE)-lined septum, labeled, and refrigerated until samples were run. Samples that did not settle after shaking were filtered through a 0.45 micron teflon membrane to remove particulates. The unused liquid portion of the original 40 mL sample was discarded into a properly labeled hazardous waste collection container and the solid portion into a separate container for proper disposal through the Defense Reutilization and Marketing Service (DRMS) or equivalent with a DRMS Form 1930, "Hazardous Waste Profile Sheet."

### Performance of Analyses

A precision biosilicate glass syringe was used to extract 100  $\mu$ L of the 1.5 mL sample. The extracted amount was added to a glass sampler syringe containing 4.9 mL of distilled water. An individual sample was injected from the sampler syringe into one

of the sample tubes, the sampler syringe was rinsed thoroughly with water and methanol, and the process was repeated until each of the 16 tubes contained a sample.

Run time for an individual sample was approximately 80 min, which included Gas Chromatograph (GC) set up and Method 1 analysis (Table 7). The sampler processed up to 16 samples, in sequence, requiring approximately 20 to 24 h to complete a batch. Unused extracts were stored at 5 °C in case additional analyses were necessary.

All laboratory supplies that came in contact with the contaminated soil sample during the analyses were rinsed thoroughly with methanol and water and then washed with detergent and water. All other supplies were washed thoroughly with detergent and water.

### Determination of Concentrations

To determine the concentration ( $\text{mg kg}^{-1}$ ) of the contaminants, linear regression analyses were performed on standards of each contaminant. For example, standards of 0, 1, 5, and 10 ppm of toluene were made and analyzed by the GC (Table 8). For each peak, the integrator printed out an area proportional to concentration of a particulate contaminant. Sample concentration was determined from the following formula, which was based on the relationship between area and concentration for authentic standards.

$$\text{concentration} = (\text{area}) \times \text{slope of line} + \text{y-intercept}$$

For example, intercept for toluene was typically 0.11 and the slope was 7.564E-05 (Figure 8). The formula then was put into a spreadsheet as follows:

$$\text{concentration} = (\text{area}) \times 7.564\text{E-}05 + 0.1077$$

**Note: A linear regression analysis on the standards should be performed regularly because areas may change through time.**

### Graphing and Analysis Results

Graphs of concentration versus time were plotted for windrow envelopes and treatment piles, and a statistical analysis was performed on Lake Shelbyville data.

Statistical analysis was not performed at the Carlyle Lake and Rend Lake sites because of insufficient replication. At least two windrows and two treatment piles are required for statistical analysis. At Rend and Carlyle, only one treatment pile existed at each site from the beginning of treatment.

**Table 7. Gas chromatograph setup and Tekmar LSC 2000 setup for Method 1.**

GC Setup	
Oven	35 °C
Internal temperature	35 °C
Initial time	4.0 minutes
Rate	4.0 degree/minute
Final value	190 °C
Final time	0.000
Inj A temperature	220 °C
Inj B temperature	200 °C
Det A temperature	270 °C
Det B temperature	47 °C
Equib time	1.00 minute
Flow A	55.7 He
	42 N2 going down
	41 H2 going down
Tekmar LSC 2000 Setup, Method 1	
Standby	35 °C
GC cycle	NI
Preheat	NI
Purge	10.00 minute
Turbo cool	NI
Prepurge	NI
Sample	NI
Dry purge	0.00
MCM desorb	Cooled to 0 °C
Cryo cooldown	NI
Desorb preheat	175 °C
Desorb	4.00 min at 180 °C
Inject	NI min at NI
Bake	8.00 min at 225 °C
BGB	OFF BGB delay: 120 seconds
Auto drain	ON
Valve	100*
Mount	100*
2016 valve	100*
2032 valve	NI
Line	100*
Heater	NI
LINE	100*
LINE	NI
Cryo union	NI
MCM bake	Heated to 90 °C*
Runs per sample	1
Bake out	OFF

NI = not included

For the Lake Shelbyville statistical analysis, two parameters were run. Times of treatment were compared, and then concentrations were compared. Significant differences were found when time was run, but NOT when concentrations were compared. Concentrations should have been essentially the same, but treatment time differed because windrow envelopes reached treatment goals faster than treatment piles.

#### ***Graphs of Concentrations vs Time***

Graphs of concentration versus time were plotted for those windrow envelopes and treatment piles that were constructed out of

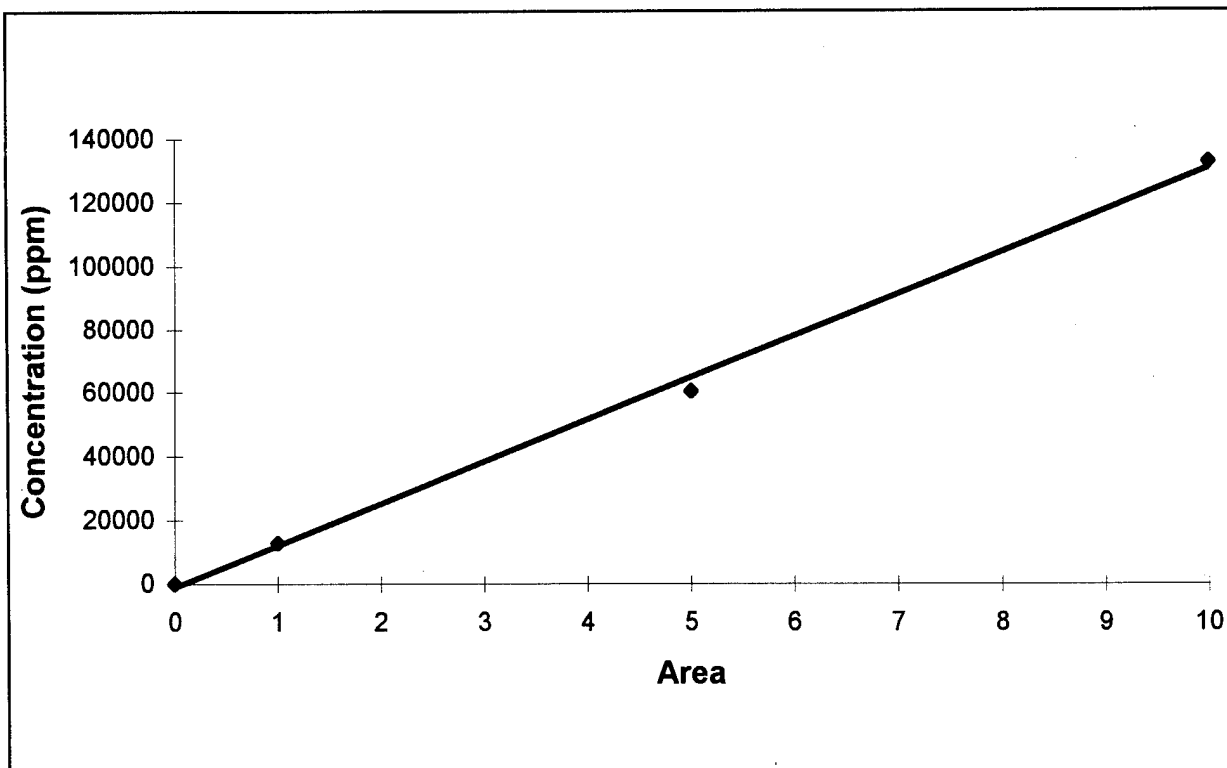
**Table 8. Example regression analysis performed on standards of toluene.**

#### **New Standards**

Conc. (ppm)	Area
0	0
1	12867
5	60295
10	132669

#### **Regression Output**

Constant	0.107731
Std Err of Y Est	0.27248
R Squared	0.997605
No. of Observations	4
Degrees of Freedom	2
X Coefficient(s)	7.564E-05
Std Err of Coef.	2.6207E-06



**Figure 8. Concentration of toluene versus GC output area.**

the same stockpiled soil at the beginning of treatment. Average benzene, toluene, ethylbenzene, xylene (BTEX) concentrations were plotted (an average of two to five samples). Next, exponential curves were fitted to points on each graph. Almost all figures display an exponential decay relationship of concentration over time. Large concentrations of the contaminant existed at the beginning and gradually decreased to zero. Scattering of points was most likely due to piles that were not uniformly contaminated. Pockets of greater contamination (such as clay-rich soil) were scattered in different areas of the pile. In addition, samples were not at the same location every week. Nevertheless, most graphs followed the same general exponential decay pattern (Appendix A).

### ***Statistical Analysis for Lake Shelbyville***

Statistical analysis was performed on Lake Shelbyville data in order to compare time for treatment of windrow envelopes with treatment piles. Windrow envelopes 1 and 2 were compared with treatment piles 1 and 2. The statistical analysis program was prepared by Dr. German Bollero of the Department of Crop Sciences, University of Illinois at Urbana-Champaign. Analysis was conducted with the general linear models procedure of the Statistical Analysis System\* (Appendix B). Results showed that the mean time to clean up a treatment pile was about 190 days, whereas the mean time to clean up a windrow was 46 days. A statistical comparison (F value) indicates that the probability that these values are significant is 99.99 percent. Therefore, windrow envelopes take a significantly shorter period of time than treatment piles for removal of POL, probably because of enhanced microbial degradation through increased aeration. This faster decomposition of POLs in windrows was also observed at Rend and Carlyle lakes.

### ***Soil Turning***

At Lake Shelbyville, windrow envelopes 3 and 4 were turned the first week and windrow envelope 4 was turned weekly thereafter. Soil in windrow envelopes 3 and 4 reached treatment goals sooner and was ready to be moved 3 weeks earlier than soil in windrow envelopes 1 and 2, which was not turned. Windrow envelope 3 was turned weekly at Rend Lake whereas 1 and 2 were not turned (Table 6). Soil treated in all three windrows was removed at the same time, but soil in windrow 3 began with a BTEX reading nearly three times greater than that in either windrow 1 or 2. Reduction of the contamination level in windrow envelope 3 occurred more rapidly than in windrows 1 and 2.

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\* SAS Institute, Inc., Cary, NC.

Data at Carlyle Lake was a combination of findings made at Rend Lake and Lake Shelbyville. Both the turned windrow envelope 1 and the unturned windrow envelope 3 reached clean up objectives for removal at the same time, but the beginning level of contamination was larger in windrow envelope 1 (Table 5). Although the beginning level of contamination in unturned windrow envelope 2 was not as large as turned windrow envelope 1, an additional 5 weeks passed before envelope 2 met state requirements.

These observations indicate that increasing aeration within the windrow envelopes by turning the soil from one side of the envelope to the other favorably affected the rate of contaminant removal.

### ***Soil Temperature and Cover Color***

Air temperature and temperature readings of soil undergoing treatment were usually made once a week or whenever soil sampling was done (Tables 9, 10, and 11). Air temperatures during the study at the three sites ranged from 13 °C recorded at Lake Shelbyville on 27 October 1994 to 34 °C recorded at Lake Shelbyville on 16 June 1994 (Table 9).

Surface temperatures of soil being treated ranged from 11 °C at Lake Shelbyville on 27 October 1994 to 38 °C at Rend Lake on 19 July 1994. Temperatures also were taken at approximately 6-in. depths in the windrow envelopes and treatment piles. Internal temperatures ranged from 12 °C at Lake Shelbyville on 27 October 1994 to 40 °C at Rend Lake on 5 July 1994 (Table 10).

According to research literature, temperatures for maximum biodegradation range between 20 and 30 °C (Dibble and Bartha 1979). Average daily temperature readings from black windrow envelopes and black-covered treatment piles were greater than white windrow envelopes and white-covered treatment piles (Figure 9). Using white-covered treatment piles and white windrow envelopes resulted in temperature readings within the range for maximum biodegradation with only two exceptions. In contrast, temperature readings for black-covered piles and windrow envelopes exceeded the range for maximum biodegradation five times. White-covered soil averaged 2 °C cooler than black-covered soil.

For biodegradation sites in Illinois during spring and summer months, white covers and windrow envelopes are more likely to enhance the biodegradation process. Locations with sufficiently cooler climates would use black covers on piles and black windrow envelopes to optimize soil temperature for biodegradation. Black could also be used to extend biodegradation efficiency to earlier in the spring, later in the fall, or, in some areas, throughout the winter.

Table 9. Temperature readings (°C) at Lake Shelbyville.

	6/10/94	6/16/94	6/24/94	7/1/94	8/8/94	8/22/94	8/29/94	9/5/94	9/12/94	10/27/94
WR1 (black)	24	33	27	27	32					
WR1 (surface)					36					
WR2 (black)	26	33	27	27	32					
WR2 (surface)					37					
WR3 (black)						31	28	27	28	
WR3 (surface)						36	29	28	30	
WR4 (black)						32	27	28	29	
WR4 (surface)						36	28	27	30	
TP 1 (center)	26	30	27	27	29	27	27	31	27	12
TP 1 (edge)	21	28								
TP 1 (surface)						30	27	25	28	12
TP 2 (center)	22	28	27	27	29	28	27	27	28	
TP 2 (edge)	22	28								
TP 2 (surface)						29	27	27	29	
TP 3 (center)										11
TP 3 (surface)										13
SP		37			36	33	31		27	13
SP (surface)									30	14
AIR	27	34	26	29	32	27	26	22	27	13

WR - windrow  
TP - treatment pile  
SP - stock pile



Table 10. Temperature readings (°C) at Rend Lake.

	6/8/94	6/15/94	6/21/94	6/28/94	7/5/94	7/12/94	7/19/94	7/26/94	8/2/94	8/9/94	8/30/94
WR1 (white)	22	27	29	27	31	27	30	27	27	27	
WR1 (surface)				28		30	33	28	32	28	
WR2 (white)	22	27	29	27	32	28	30	27	27	28	
WR2 (surface)				27		30	32	29	34	29	
WR3 (white)	22	27	29	26	33	27	32	27	27	28	
WR3 (surface)				26		29	33	29	33	30	
WR4 (black)			32	27	40	29	33	29			
WR4 (surface)				32		35	37	32			
WR5 (black)									27	31	26
WR5 (surface)									31	33	27
WR6 (white)											22
WR6 (surface)											24
WR7 (white)											24
WR7 (surface)											26
WR8 (white)											24
WR8 (surface)											26
TP 1 (center)	22	26	28	27	32	27	35	27	29	26	
TP 1 (surface)						30	32	28	32	26	
SP 1		29	32	28	32	32	32	32	36	31	26
SP 1 (surface)						37	38	36	38	31	27
SP2			31	27				39			
AIR	27	29	31	32	32	29	32	28	33	27	24

WR - windrow; TP - treatment pile; SP - stock pile

Table 11. Temperature readings (°C) at Carlyle Lake.

	6/1/94	6/7/94	6/14/94	6/22/94	6/29/94	7/6/94	7/13/94	7/20/94	7/27/94	8/03/94	8/10/94	8/24/94	8/31/94
WR1 (black)	25	27	27	31	27	29	28	30	21			32	26
WR1 (surface)									21			33	25
WR2 (black)	25	27	27	31	27	30	28	29	22	28	27		
WR2 (surface)									23	32	24		
WR3 (black)	25	28	29	30	27	31	28	29	22				
WR3 (surface)									21				
WR4 (white)				30	27	29	27	28	22	27	26		
WR4 (surface)									20	29	23		
WR5 (black)										27	27	31	24
WR5 (surface)										27	26	32	26
WR6 (black)										27	26	30	24
WR6 (surface)										27	26	32	26
WR7 (white)												29	23
WR7 (surface)												32	24
TP Center	25		27	30	27	27	27	29	22	28	24	30	23
TP surface									22		22	31	24
TP edge	21	26	27	27									
SP			32	36	27	33	33	33	29	33	27		
SP (surface)											22		
AIR	29	29		27	27	29	27	28	21	29	23	32	24

WR - windrow; TP - treatment pile; SP - stock pile

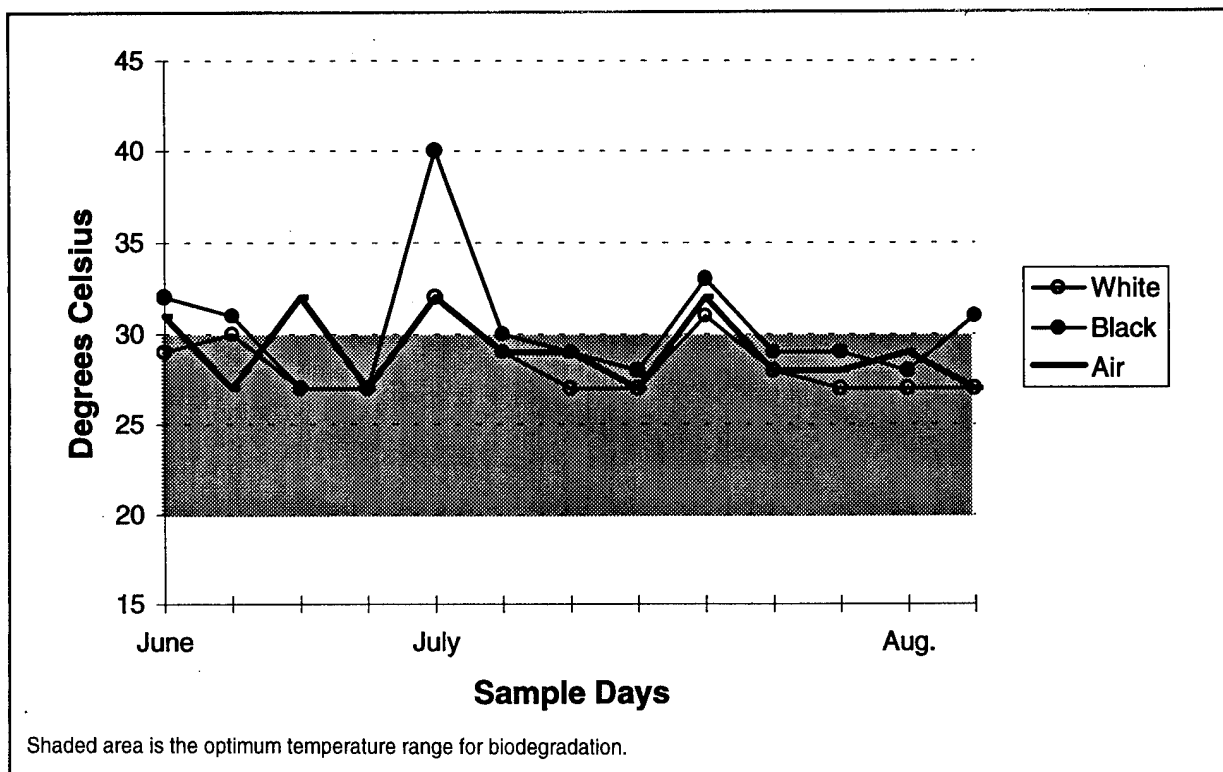


Figure 9. Plot of soil temperature for black and white covered soil relative to air temperatures.

## 6 Conclusions

Statistical analysis performed on Lake Shelbyville data showed that windrow envelopes take a significantly shorter time than treatment piles for removal of POLs. A similar, faster decomposition of POLs in windrows also was observed at Rend and Carlyle lakes.

Field observations, laboratory tests, and statistical analysis support the conclusion that treatment of POL-contaminated soil in windrow envelopes is an efficient method superior to treatment in piles.

Other results noted:

- increased aeration within windrows favorably affects the rate of contaminant removal
- for biodegradation sites in Illinois, white covers enhance the biodegradation process.

## 7 Lessons Learned

Some lessons learned, especially in the laboratory, may be too specific to have widespread interest, but others generally may be useful to avoid mistakes or improve efficiency. Laboratory equipment (Table 12) and field equipment used (Table 13) have been itemized.

### Laboratory

#### *Cryofocusing*

A typical setup for gas chromatographic analysis by purge and trap includes a cryofocusing unit requiring liquid nitrogen. The cryofocusing unit quickly cools a sample and forces it through the column for a quicker result. Cryofocusing consumes liquid nitrogen and adds noise and maintenance issues. Researchers determined that cryofocusing was not necessary for this application. Direct injection was used instead. This method saved money, time, and space.

#### *Analysis Standards*

To analyze data, standards were purchased and analyzed to acquire parameters for contaminants involved. Original standards were combined BTEX and naphthalene along with methyl tert-butylether and trimethylbenzenes. Exact retention times were difficult to evaluate, possibly because of error from using several contaminants. Consequently, using individual standards is recommended, at least for initial retention times; combined standards can be used later for backup. When using the pure individual standard, testing of a particular substance at concentration levels the system can actually handle is most important. Main contaminants of this analysis were benzene and toluene, which have a tendency to contaminate the lines and trap of the purge and trap, which requires baking the trap. However, if only the lines are contaminated, the system can be flushed to clean them.

**Table 12. Laboratory equipment.**

Instruments Used	
Leak Detection Equipment	
Vocol Capillary Column	
Tekmar Purge and Trap	
ALS 2016 Sampler	
LSC 2000 Trap	
Hewlett Packard Gaschromotographer (GC) 5890 Series II	
Hewlett Packard Integrater	
Shaker Table	
Supply List	
4 mL vials	rubber gloves
1.5 mL vials	goggles
distilled water	syringes
GC Resolve methanol (purge and trap)	beakers
BETX/Napthalene Standards	clamps
Neat Standards (BETX/Napthalene individuals)	
Compressed Gases	
Ultra High Purity (UHP) Air	
Helium	
Hydrogen	

**Table 13. Field equipment.**

Sampling Supplies	
4 ounce mason jars	spade
gloves	labels
mixing container	log books
shovel	rakes
plastic liners	tarpaulins
straw bales	sandbags
Soil Moving	
End Loader/Back Hoe	
Dump Truck (optional)	

**Sampling Syringe**

Using a plastic sampling syringe may create analysis error because of a contaminant's tendency to sorb onto the plastic and rubber. Investigation demonstrated no sorption in this particular study, but this tendency should be considered in this type of analysis.

**Temperature Adjustments**

Seasonal changes may require adjustment of the purge and trap to room temperature. Therefore, parameters such as sam-

ple temperature may have to be adjusted throughout analysis, rerunning standards to adjust data accordingly. When temperatures change, standards should be rerun to adjust retention times in the analysis.

## **Field**

### ***Site Selection and Preparation***

Careful site selection and preparation is essential. A water source should be close for quick and easy watering. Durable reinforced ground cloths should be used instead of other cheaper plastic, which becomes brittle and cracks. Before soil is placed on reinforced ground cloths, the underlying surface should be reasonably level and smooth. This is especially important when soil is turned during the treatment process and when it is later moved off the plastic. If reinforced ground cloths are to be reused, do not place them on concrete or asphalt. Although these surfaces are smooth, the plastic tears easily on these surfaces.

### ***Recordkeeping***

Accurate, timely, and complete records are essential when soil is turned, fertilized, or watered, as well as all sampling dates and locations.

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- Pramer, D., and R. Bartha, "Preparation and Processing of Soil Samples for Biodegradation Studies," *Environmental Letters*, vol 2 (1972), pp 217-224.



## Appendix A: Lake Site Remediation Patterns

Lake Shelbyville

BTEX Concentrations (averages)

DOT = Days of Treatment

WR1

DOT	BTEX
11	13.147
25	8.464
31	0.000
39	0.706
46	0.570
53	0.193

WR 3

DOT	BTEX
0	6.238
7	1.616
14	0.001
21	0.245
28	0.246
35	0.216

WR2

DOT	BTEX
11	29.720
25	12.801
31	4.829
39	1.630
46	0.451
53	4.926

WR4

DOT	BTEX
0	19.260
7	4.892
14	0.009
21	0.416
28	1.703
35	0.467
53	0.446

TP1

DOT	BTEX
11	8.832
25	13.017
31	20.153
39	16.548
46	5.663
53	7.392
60	10.264
67	8.863
74	0.000
81	31.183
88	0.276
95	0.022
109	7.929
116	0.157
129	0.537
136	5.409
143	0.167
150	0.000
157	1.516
164	0.305
185	0.672
206	0.000

TP2

DOT	BTEX
11	0.157
25	8.056
31	4.340
39	0.279
46	7.888
53	0.612
60	1.796
74	0.000
81	0.990
88	0.125
95	0.189
109	1.293
116	2.544
129	0.196
136	0.634
143	2.331
150	0.197
157	0.360
164	0.173
185	0.798
198	0.000
206	0.000

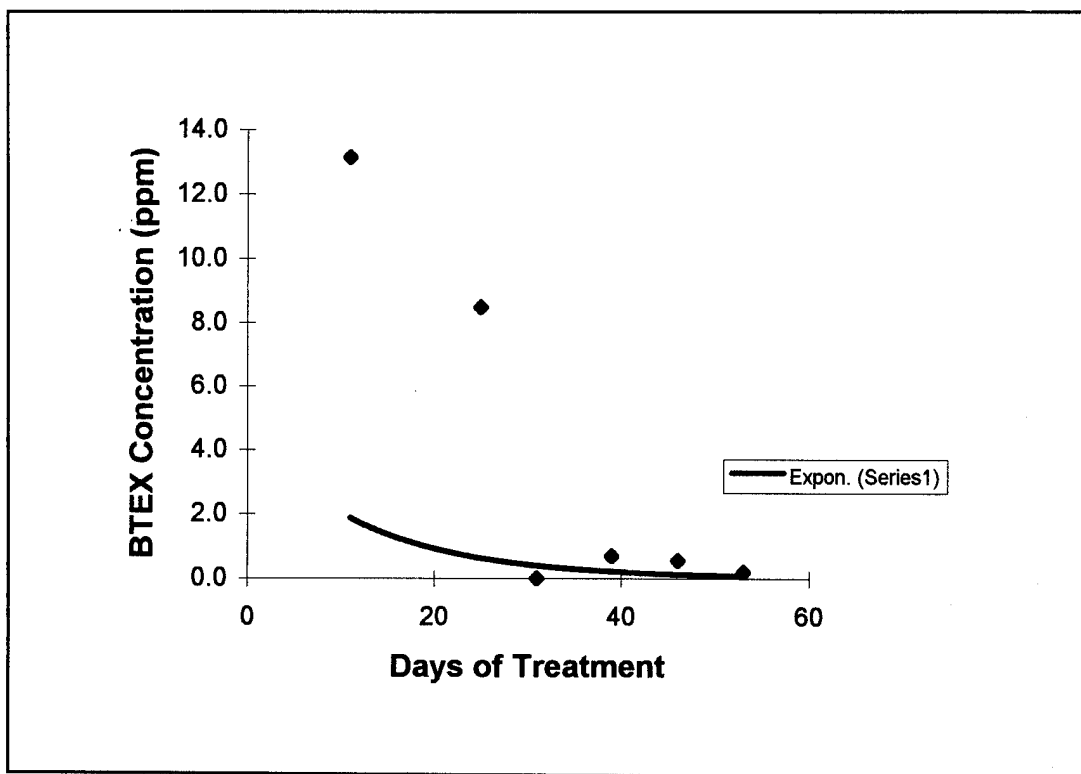


Figure A1. Remediation pattern of Windrow 1 (WR1), Lake Shelbyville.

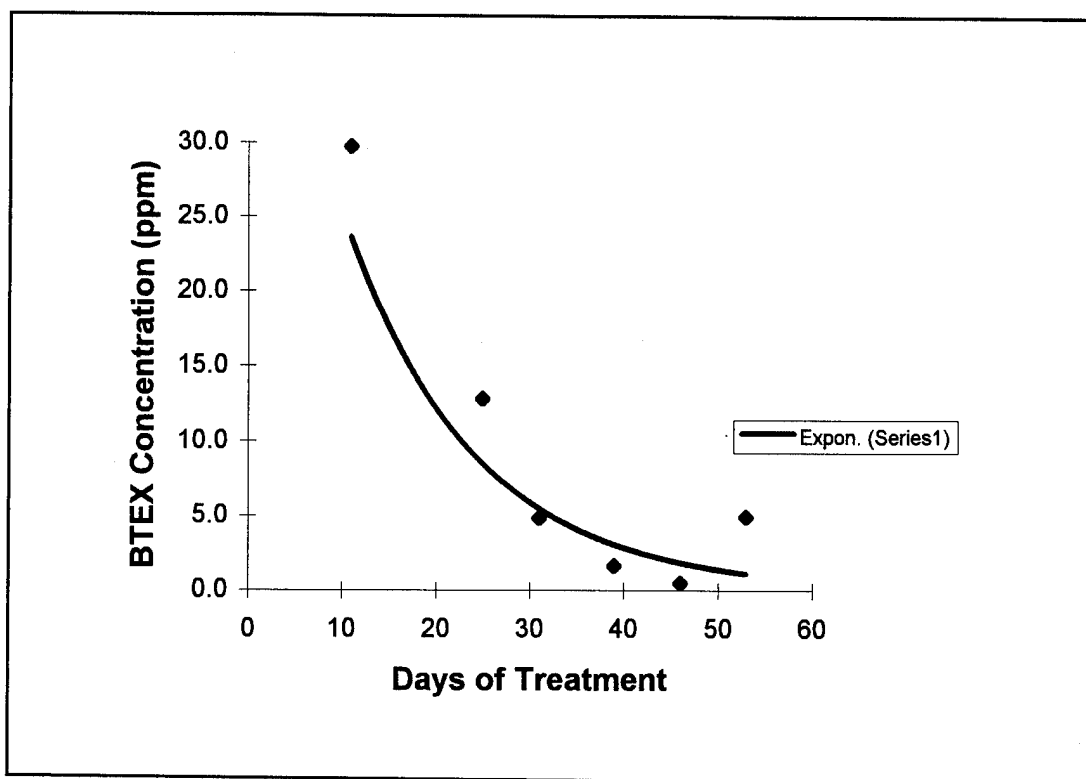


Figure A2. Remediation pattern of WR2, Lake Shelbyville.

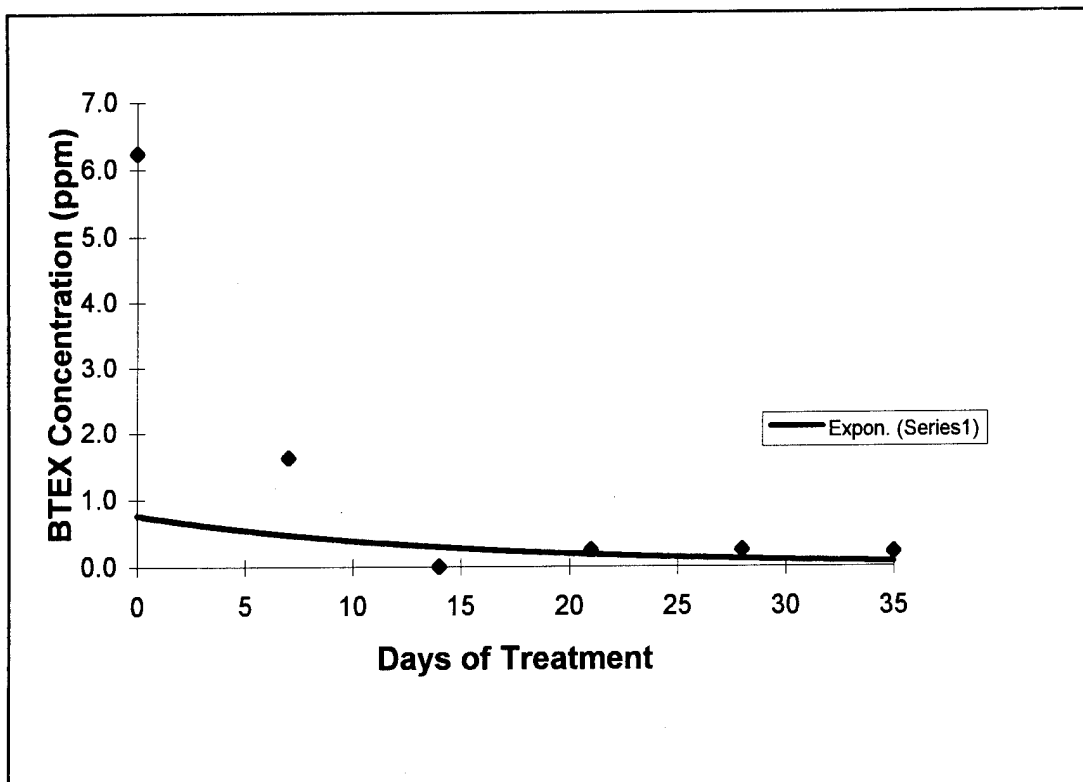


Figure A3. Remediation pattern of WR3, Lake Shelbyville.

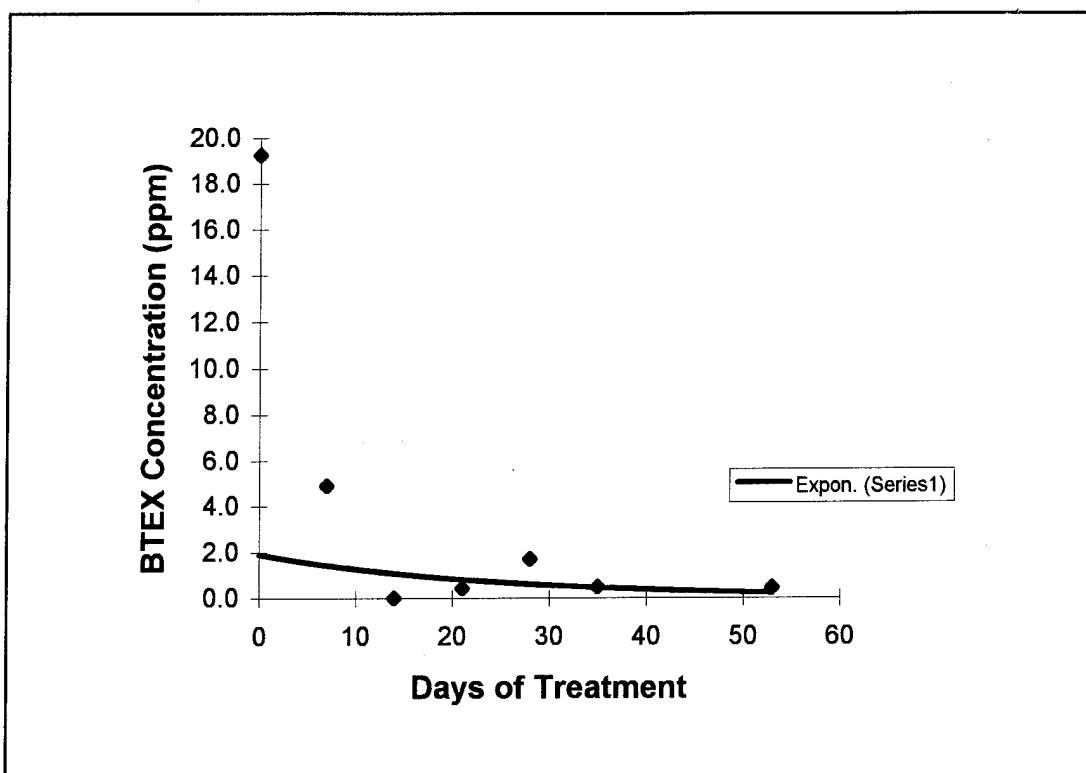


Figure A4. Remediation pattern of WR4, Lake Shelbyville.

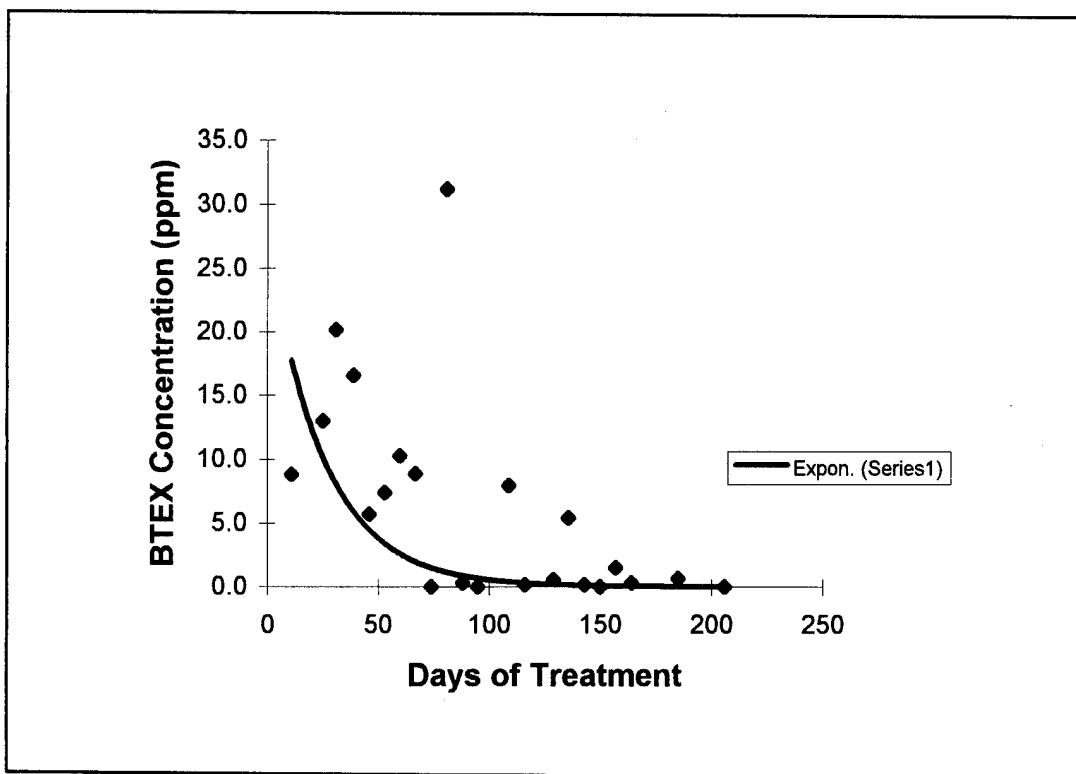


Figure A5. Remediation pattern of Treatment Pile 1 (TP1), Lake Shelbyville.

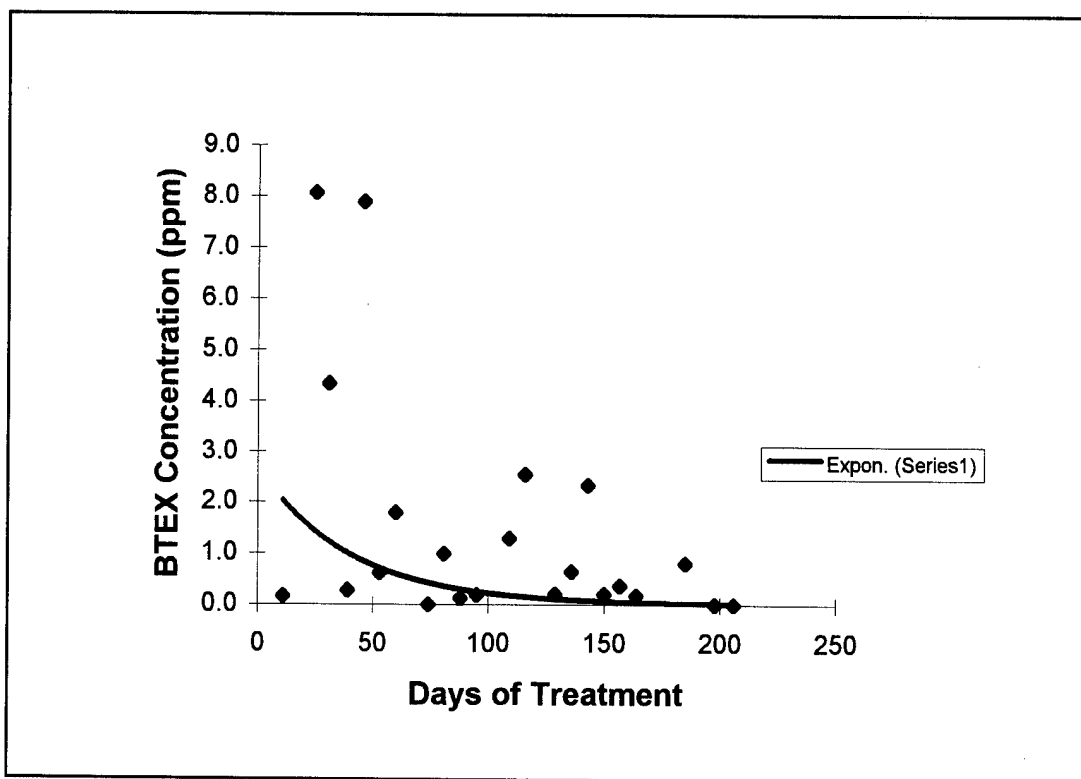


Figure A6. Remediation pattern of TP2, Lake Shelbyville.

## Lake Carlyle

## BTEX Concentrations (averages)

DOT = Days of Treatment

## WR1

DOT	BTEX
0	0.573
13	0.135
19	0.312
26	0.194
34	0.068
41	0.452
48	0.021
55	0.405

## WR3

DOT	BTEX
13	0.097
19	0.658
26	0.108
34	0.386
41	0.043
48	0.035
55	0.216

## WR2

DOT	BTEX
19	0.443
26	0.251
34	0.363
41	0.000
48	0.000
55	0.528
62	2.023
69	3.082
77	0.459
83	0.395
90	0.206
97	0.258
105	0.184

## TP

DOT	BTEX
19	1.033
26	8.330
34	0.317
41	2.850
48	0.694
55	0.313
69	1.174
83	0.160
90	0.000
105	0.000
124	0.000
131	2.788
145	0.073
152	0.036

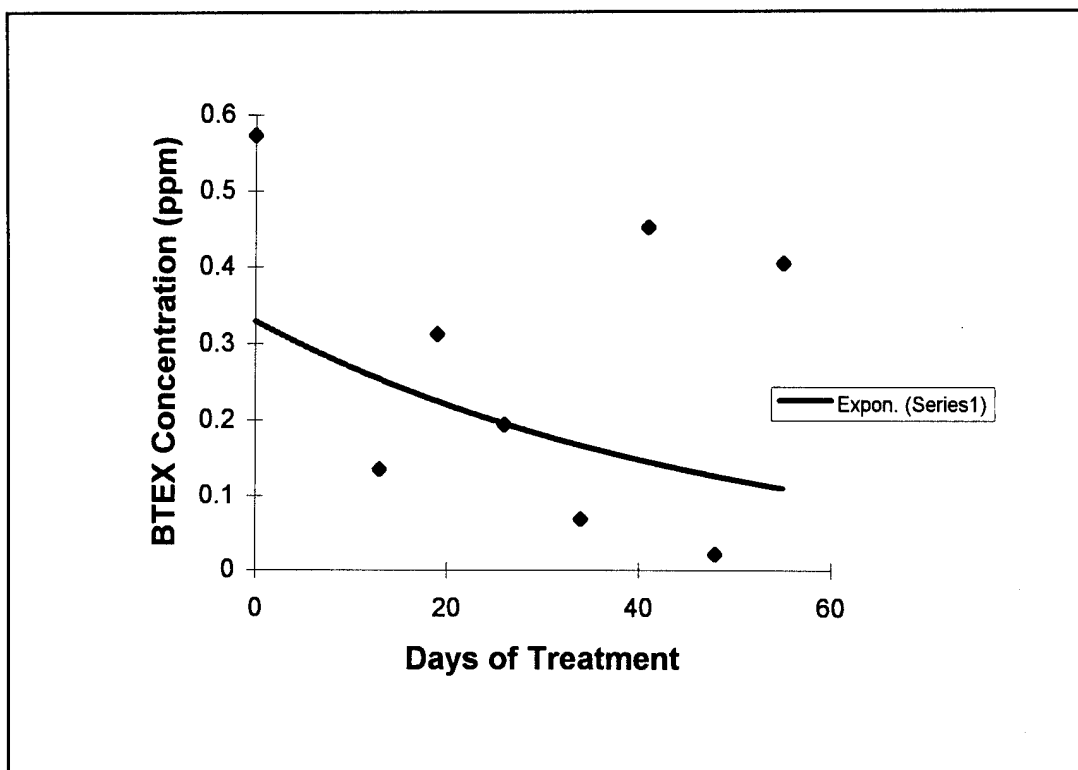


Figure A7. Remediation pattern of WR1, Carlyle Lake.

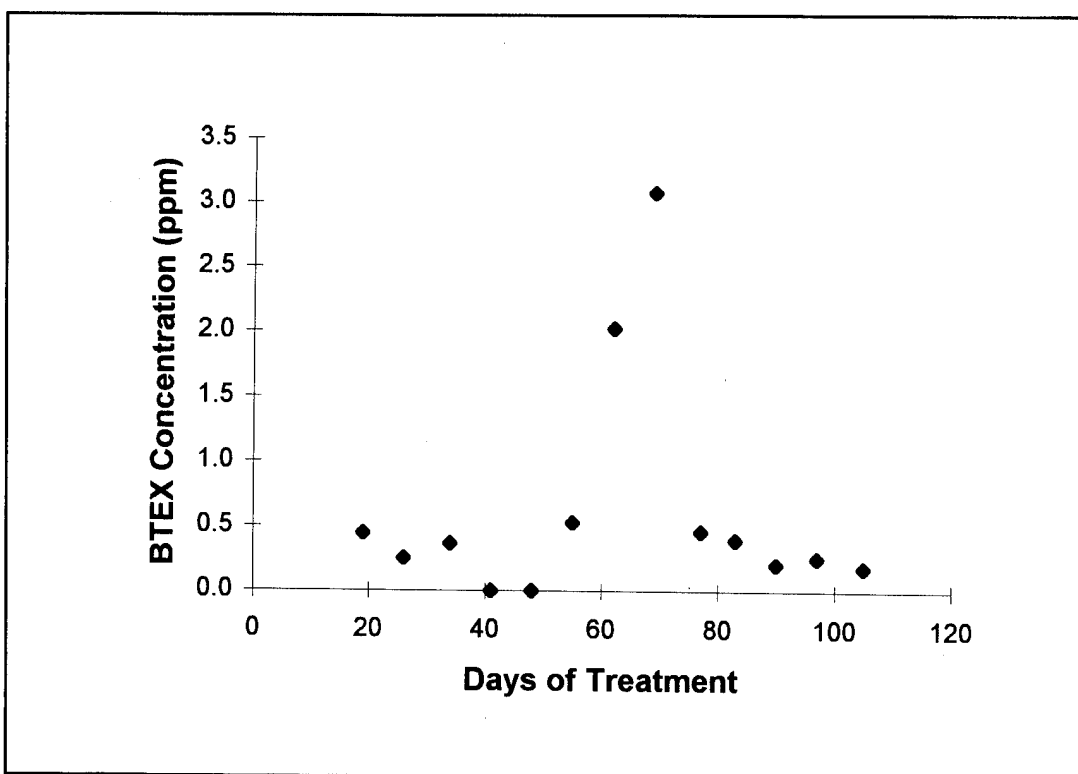


Figure A8. Remediation pattern of WR2, Carlyle Lake.

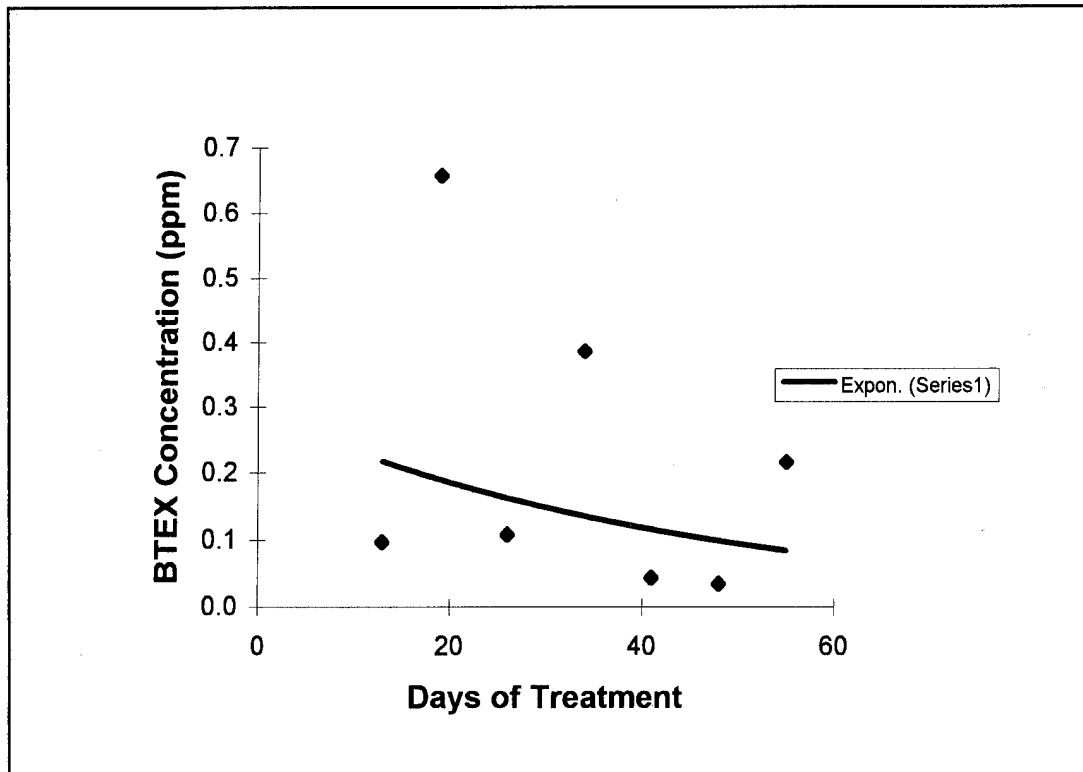


Figure A9. Remediation pattern of WR3, Carlyle Lake.

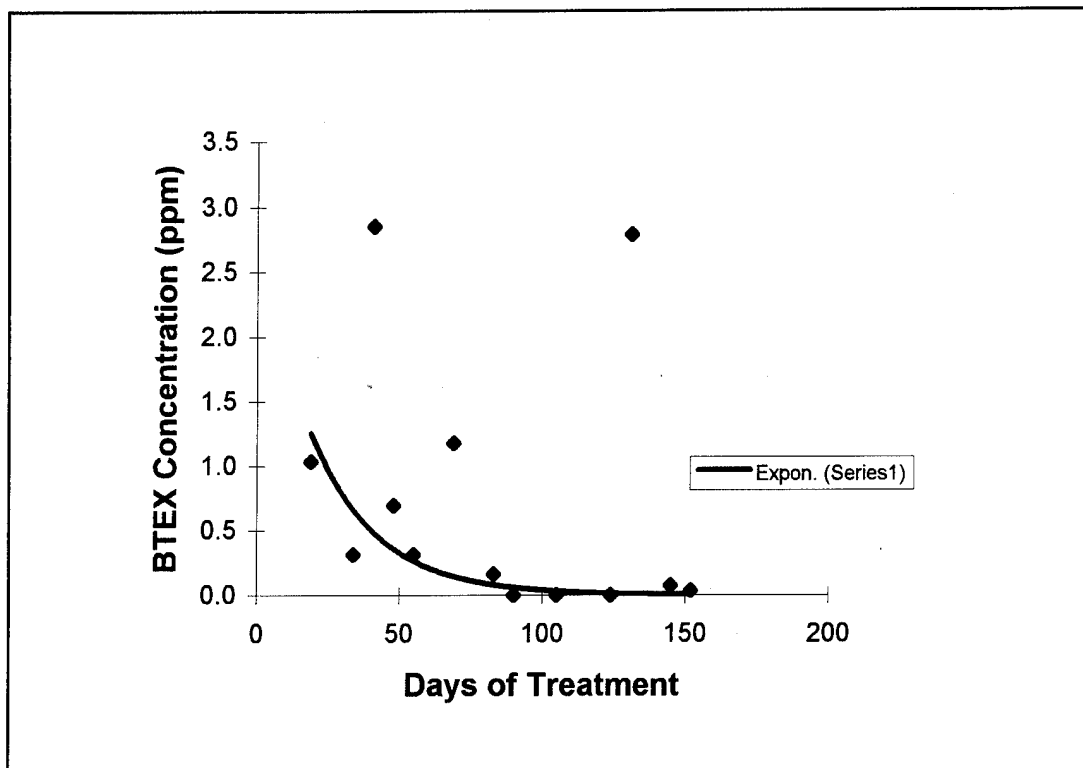


Figure A10. Remediation pattern of treatment pile, Carlyle Lake.



## Lake Rend

## BTEX Concentrations (averages)

DOT = Days of Treatment

## WR1

DOT	BTEX
0	1.823
7	1.659
13	0.135
20	5.031
26	1.007
33	1.913
40	0.735
47	0.480
54	6.092
61	0.026
69	0.218
75	0.014
82	0.010

## WR3

DOT	BTEX
1	5.000
13	0.832
20	1.163
26	0.150
33	0.015
40	0.000
47	0.104
54	0.626
61	0.677
75	0.005
82	0.000

## WR2

DOT	BTEX
1	1.531
13	1.054
20	1.739
26	3.497
33	0.784
40	0.003
47	0.121
54	0.886
61	0.271
69	0.118
75	0.000
82	0.203

## TP1

DOT	BTEX
1	1.140
13	0.294
20	0.010
26	0.160
33	0.135
40	0.013
47	0.098
61	0.256
69	0.833
75	0.025
82	0.000

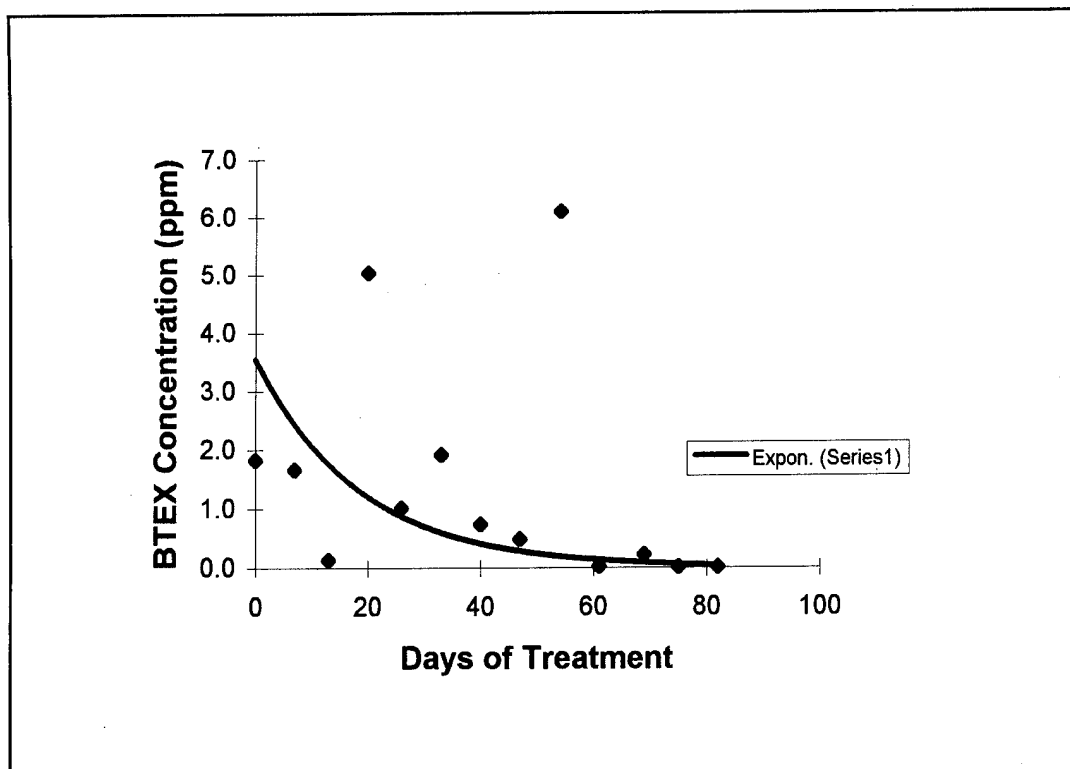


Figure A11. Remediation pattern of WR1, Rend Lake.

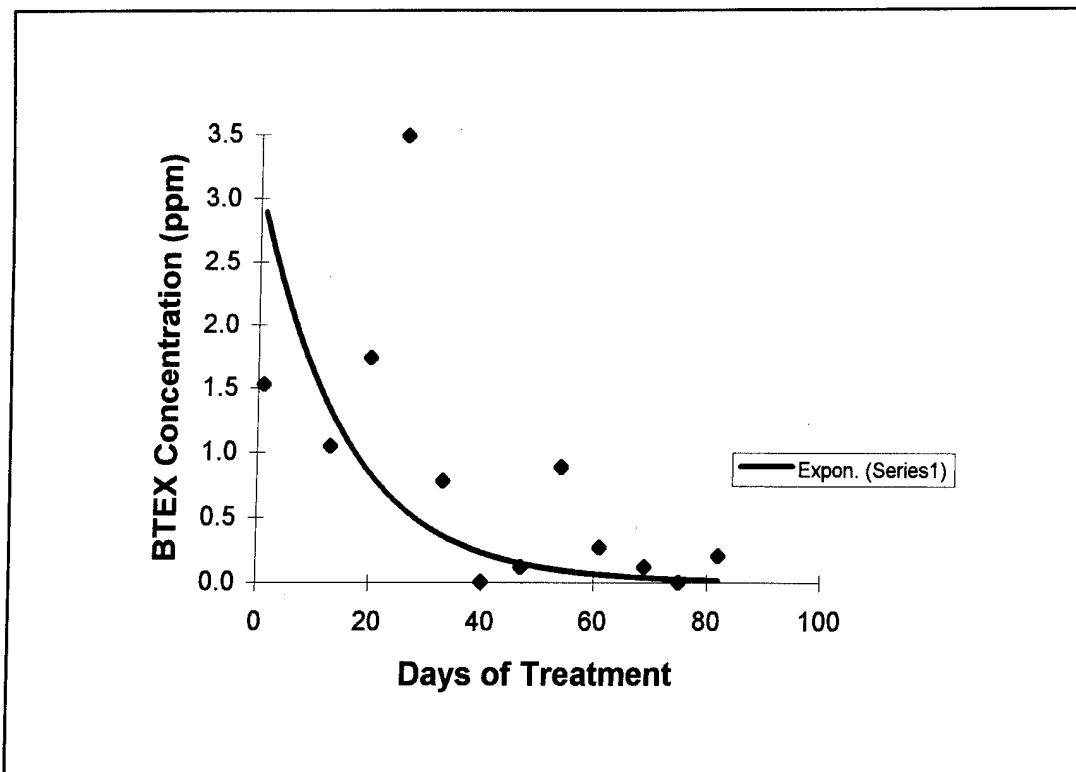


Figure A12. Remediation pattern of WR2, Rend Lake.

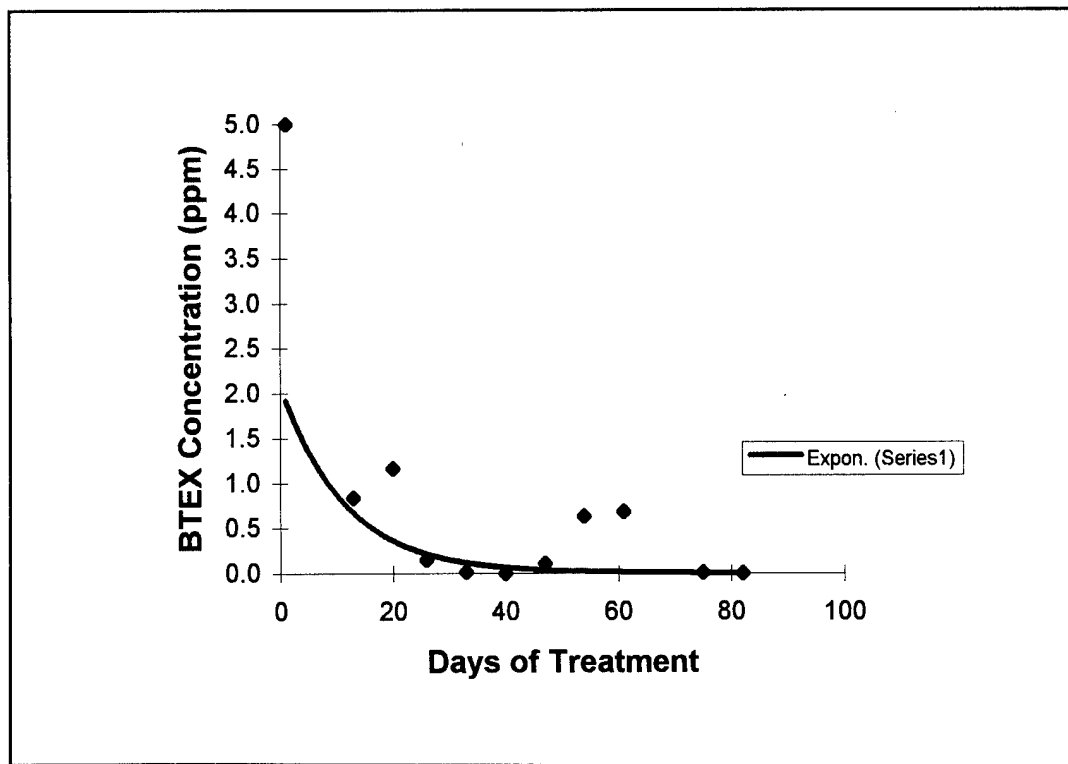


Figure A13. Remediation pattern of WR3, Rend Lake.

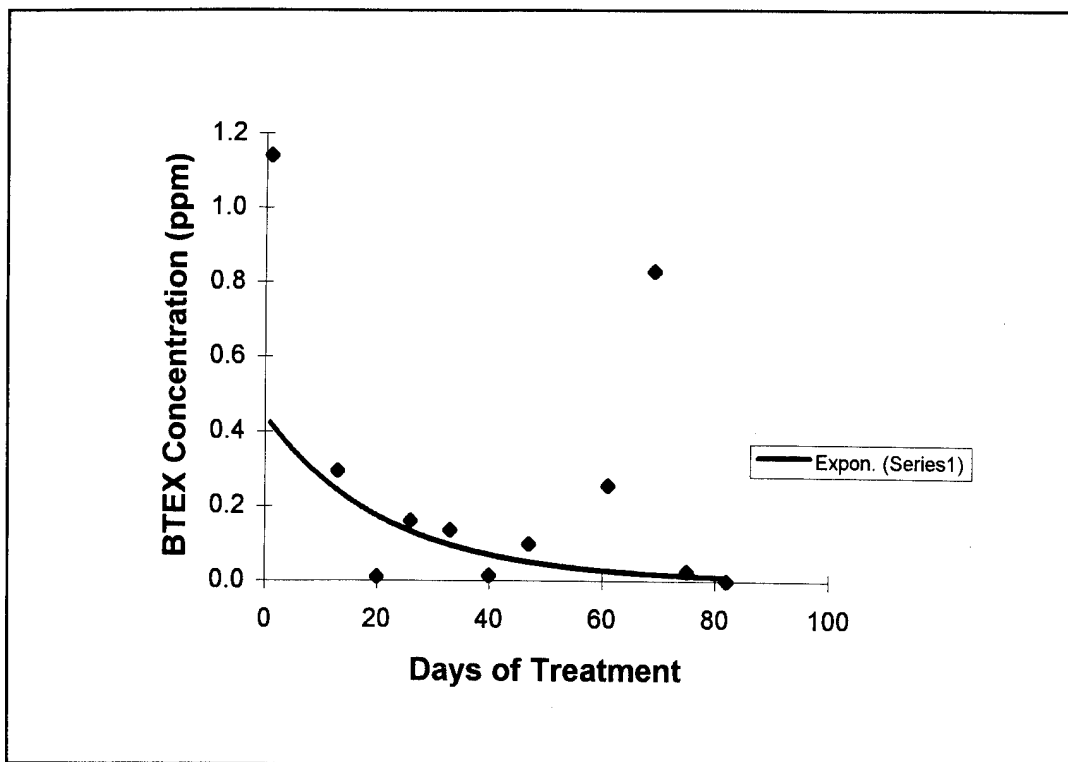


Figure A14. Remediation pattern of TP1, Rend Lake.

## Appendix B: Statistical Analysis System Program Output

```
options ls=74 ps=1500;
data eva;
infile 'd:\german\eva\stats.prn';
input Treat$ Rep Day Sample Date Conc;

proc print;

*****ANOVA comparing treat*****;
proc glm;
class treat rep sample;
model date= treat;
means treat;

*****;
data eval;
infile 'd:\german\eva\stats2.prn';
input Treat$ Rep Sample C11 C25 C31 C39 C46 C53
C60 C74 C81 C88 C95 C109 C116 C129 C136 C143 C150 C157 C164 C185;

proc print;

*****TIME SERIES*****;

proc glm;
class treat rep sample;
model C11 C25 C31 C39 C46 C53
C60 C74 C81 C88 C95 C109 C116
C129 C136 C143 C150 C157 C164 C185= treat/NOUNI;
REPEATED day 20 /summary;

run;
```

The SAS System 1  
11:32 Tuesday, November 7, 1995

OBS	TREAT	REP	DAY	SAMPLE	DATE	CONC
1	WR	1	11	1	39	0.5890
2	WR	1	11	2	39	7.1320
3	WR	1	11	3	39	5.4260
4	WR	1	25	1	39	1.1390
5	WR	1	25	2	39	3.0580
6	WR	1	25	3	39	4.2670
7	WR	1	31	1	39	0.0000
8	WR	1	31	2	39	0.0000
9	WR	1	39	1	39	0.7060
10	WR	1	39	2	39	0.0000
11	WR	1	39	3	39	0.0000
12	WR	1	46	1	39	0.3255
13	WR	1	46	2	39	0.2442
14	WR	1	53	1	39	0.0677
15	WR	1	53	2	39	0.0635
16	WR	1	53	3	39	0.0620
17	WR	1	60	1	39	0.0000
18	WR	1	60	2	39	0.0000
19	WR	1	74	1	39	0.0000
20	WR	1	74	2	39	0.0000
21	WR	1	81	1	39	0.0000
22	WR	1	81	2	39	0.0000
23	WR	1	88	1	39	0.0000
24	WR	1	88	2	39	0.0000
25	WR	1	95	1	39	0.0000
26	WR	1	95	2	39	0.0000
27	WR	1	109	1	39	0.0000
28	WR	1	109	2	39	0.0000
29	WR	1	116	1	39	0.0000
30	WR	1	116	2	39	0.0000
31	WR	1	129	1	39	0.0000
32	WR	1	129	2	39	0.0000
33	WR	1	129	3	39	0.0000
34	WR	1	136	1	39	0.0000
35	WR	1	136	2	39	0.0000
36	WR	1	143	1	39	0.0000
37	WR	1	143	2	39	0.0000
38	WR	1	150	1	39	0.0000
39	WR	1	150	2	39	0.0000
40	WR	1	150	3	39	0.0000
41	WR	1	157	1	39	0.0000
42	WR	1	157	2	39	0.0000
43	WR	1	157	3	39	0.0000
44	WR	1	164	1	39	0.0000
45	WR	1	164	2	39	0.0000
46	WR	1	185	1	39	0.0000
47	WR	1	185	2	39	0.0000
48	WR	2	11	1	53	3.8465
49	WR	2	11	2	53	18.2514
50	WR	2	11	3	53	7.6223
51	WR	2	25	1	53	6.2894
52	WR	2	25	2	53	3.7550
53	WR	2	25	3	53	2.7560
54	WR	2	31	1	53	0.0000
55	WR	2	31	2	53	3.0358
56	WR	2	39	1	53	1.1251
57	WR	2	39	2	53	0.5050
58	WR	2	39	3	53	0.0000
59	WR	2	46	1	53	0.2413
60	WR	2	46	2	53	0.2100
61	WR	2	53	1	53	3.6380
62	WR	2	53	2	53	1.1672

63	WR	2	53	3	53	0.1203
64	WR	2	60	1	53	0.0000
65	WR	2	60	2	53	0.0000
66	WR	2	74	1	53	0.0000
67	WR	2	74	2	53	0.0000
68	WR	2	81	1	53	0.0000
69	WR	2	81	2	53	0.0000
70	WR	2	88	1	53	0.0000
71	WR	2	88	2	53	0.0000
72	WR	2	95	1	53	0.0000
73	WR	2	95	2	53	0.0000
74	WR	2	109	1	53	0.0000
75	WR	2	109	2	53	0.0000
76	WR	2	116	1	53	0.0000
77	WR	2	116	2	53	0.0000
78	WR	2	129	1	53	0.0000
79	WR	2	129	2	53	0.0000
80	WR	2	129	3	53	0.0000
81	WR	2	136	1	53	0.0000
82	WR	2	136	2	53	0.0000
83	WR	2	143	1	53	0.0000
84	WR	2	143	2	53	0.0000
85	WR	2	150	1	53	0.0000
86	WR	2	150	2	53	0.0000
87	WR	2	150	3	53	0.0000
88	WR	2	157	1	53	0.0000
89	WR	2	157	2	53	0.0000
90	WR	2	157	3	53	0.0000
91	WR	2	164	1	53	0.0000
92	WR	2	164	2	53	0.0000
93	WR	2	185	1	53	0.0000
94	WR	2	185	2	53	0.0000
95	TP	1	11	1	198	15.0351
96	TP	1	11	2	198	8.8971
97	TP	1	11	3	198	2.5651
98	TP	1	25	1	198	4.8833
99	TP	1	25	2	198	9.7939
100	TP	1	25	3	198	24.3730
101	TP	1	31	1	198	29.4050
102	TP	1	31	2	198	10.9050
103	TP	1	39	1	198	8.8425
104	TP	1	39	2	198	38.2390
105	TP	1	39	3	198	2.5631
106	TP	1	46	1	198	8.9530
107	TP	1	46	2	198	7.1800
108	TP	1	53	1	198	0.2745
109	TP	1	53	2	198	21.5020
110	TP	1	53	3	198	0.3994
111	TP	1	60	1	198	9.1860
112	TP	1	60	2	198	11.3405
113	TP	1	74	1	198	0.0000
114	TP	1	74	2	198	0.0000
115	TP	1	81	1	198	13.8931
116	TP	1	81	2	198	48.4730
117	TP	1	88	1	198	0.0000
118	TP	1	88	2	198	0.5522
119	TP	1	95	1	198	0.0000
120	TP	1	95	2	198	0.0432
121	TP	1	109	1	198	1.6589
122	TP	1	109	2	198	14.2000
123	TP	1	116	1	198	0.0811
124	TP	1	116	2	198	0.3898
125	TP	1	129	1	198	1.3915
126	TP	1	129	2	198	0.2186
127	TP	1	129	3	198	0.0000
128	TP	1	136	1	198	7.3210
129	TP	1	136	2	198	3.4977

130	TP	1	143	3	198	0.3346
131	TP	1	143	1	198	0.0000
132	TP	1	150	2	198	0.0000
133	TP	1	150	1	198	0.0000
134	TP	1	150	2	198	0.0000
135	TP	1	157	1	198	1.1199
136	TP	1	157	2	198	1.9115
137	TP	1	157	3	198	0.0206
138	TP	1	164	1	198	0.0124
139	TP	1	164	2	198	0.5973
140	TP	1	185	1	198	0.0000
141	TP	1	185	2	198	1.3436
142	TP	2	11	1	185	0.4697
143	TP	2	11	2	185	0.0000
144	TP	2	11	3	185	0.0000
145	TP	2	25	1	185	3.2633
146	TP	2	25	2	185	3.5493
147	TP	2	25	3	185	14.4100
148	TP	2	31	1	185	9.4518
149	TP	2	31	2	185	0.0358
150	TP	2	39	1	185	0.7393
151	TP	2	39	2	185	0.0023
152	TP	2	39	3	185	0.0959
153	TP	2	46	1	185	0.4506
154	TP	2	46	2	185	9.3858
155	TP	2	53	1	185	0.0883
156	TP	2	53	2	185	1.4581
157	TP	2	53	3	185	0.2886
158	TP	2	60	1	185	0.0893
159	TP	2	60	2	185	3.5020
160	TP	2	74	1	185	0.0000
161	TP	2	74	2	185	0.0000
162	TP	2	81	1	185	0.5320
163	TP	2	81	2	185	1.4480
164	TP	2	88	1	185	0.0000
165	TP	2	88	2	185	0.2493
166	TP	2	95	1	185	0.0000
167	TP	2	95	2	185	0.3771
168	TP	2	109	1	185	0.3743
169	TP	2	109	2	185	2.2122
170	TP	2	116	1	185	7.6320
171	TP	2	116	2	185	0.0000
172	TP	2	129	1	185	0.5512
173	TP	2	129	2	185	0.0380
174	TP	2	129	3	185	0.0000
175	TP	2	136	1	185	1.2690
176	TP	2	136	2	185	0.0000
177	TP	2	143	1	185	5.8940
178	TP	2	143	2	185	0.1244
179	TP	2	150	1	185	0.2740
180	TP	2	150	2	185	0.0000
181	TP	2	150	3	185	0.3165
182	TP	2	157	1	185	0.5403
183	TP	2	157	2	185	0.4534
184	TP	2	157	3	185	0.0851
185	TP	2	164	1	185	0.1611
186	TP	2	164	2	185	0.1847
187	TP	2	185	1	185	1.3510
188	TP	2	185	2	185	0.2455

The SAS System 2  
11:32 Tuesday, November 7, 1995

General Linear Models Procedure  
Class Level Information

Class	Levels	Values
TREAT	2	TP WR
REP	2	1 2
SAMPLE	3	1 2 3

Number of observations in data set = 188

The SAS System 3  
11:32 Tuesday, November 7, 1995

General Linear Models Procedure

Dependent Variable: DATE

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	1	995001.75	995001.75	21576.25	0.0001
Error	186	8577.50	46.12		
Corrected Total	187	1003579.25			

R-Square	C.V.	Root MSE	DATE Mean
0.991453	5.718607	6.7908	118.75

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TREAT	1	995001.75	995001.75	21576.25	0.0001

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TREAT	1	995001.75	995001.75	21576.25	0.0001

The SAS System 4  
11:32 Tuesday, November 7, 1995

General Linear Models Procedure

Level of		-----DATE-----	
TREAT	N	Mean	SD
TP	94	191.500000	6.53485280
WR	94	46.000000	7.03753378



The SAS System<sup>5</sup>  
11:32 Tuesday, November 7, 1995

OBS	TREAT	REP	SAMPLE	C11	C25	C31	C39	C46
1	WR	1	1	0.5890	1.1390	0.0000	0.7060	0.32550
2	WR	1	2	7.1320	3.0580	0.0000	0.0000	0.24420
3	WR	1	3	5.4260	4.2670	.	0.0000	.
4	WR	2	1	3.8465	6.2894	0.0000	1.1251	0.24130
5	WR	2	2	18.2514	3.7550	3.0358	0.5050	0.20998
6	WR	2	3	7.6223	2.7560	.	0.0000	.
7	TP	1	1	15.0351	4.8833	29.4050	8.8425	8.95300
8	TP	1	2	8.8971	9.7939	10.9050	38.2390	7.18000
9	TP	1	3	2.5651	24.3730	.	2.5631	.
10	TP	2	1	0.4697	3.2633	9.4518	0.7393	0.45065
11	TP	2	2	0.0000	3.5493	0.0358	0.0023	9.38581
12	TP	2	3	0.0000	14.4100	.	0.0959	.

OBS	C53	C60	C74	C81	C88	C95	C109	C116
1	0.0677	0.00000	0	0.0000	0.00000	0.00000	0.0000	0.00000
2	0.0635	0.00000	0	0.0000	0.00000	0.00000	0.0000	0.00000
3	0.0620	.	.	.	.	.	.	.
4	3.6380	0.00000	0	0.0000	0.00000	0.00000	0.0000	0.00000
5	1.1672	0.00000	0	0.0000	0.00000	0.00000	0.0000	0.00000
6	0.1203	.	.	.	.	.	.	.
7	0.2745	9.18600	0	13.8931	0.00000	0.00000	1.6589	0.08110
8	21.5021	1.34048	0	48.4730	0.55220	0.04320	14.2000	0.38978
9	0.3994	.	.	.	.	.	.	.
10	0.0883	0.08930	0	0.5320	0.00000	0.00000	0.3743	7.63200
11	1.4581	3.50200	0	1.4480	0.24928	0.37713	42.2122	0.00000
12	0.2886	.	.	.	.	.	.	.

OBS	C129	C136	C143	C150	C157	C164	C185
1	0.00000	0.0000	0.00000	0.0000	0.00000	0.00000	0.0000
2	0.00000	0.0000	0.00000	0.0000	0.00000	0.00000	0.0000
3	0.00000	.	.	0.0000	0.00000	.	.
4	0.00000	0.0000	0.00000	0.0000	0.00000	0.00000	0.0000
5	0.00000	0.0000	0.00000	0.0000	0.00000	0.00000	0.0000
6	0.00000	.	.	0.0000	0.00000	.	.
7	1.39150	7.3210	0.33458	0.0000	1.11989	0.01237	0.0000
8	0.21859	3.4977	0.00000	0.0000	1.91150	0.59734	1.3436
9	0.00000	.	.	0.0000	0.02059	.	.
10	0.55120	1.2690	5.89400	0.2740	0.54030	0.16110	1.3510
11	0.03800	0.0000	0.12440	0.0000	0.45340	0.18470	0.2455
12	0.00000	.	.	0.3165	0.08510	.	.

The SAS System<sup>6</sup>  
11:32 Tuesday, November 7, 1995

General Linear Models Procedure  
Class Level Information

Class	Levels	Values
TREAT	2	TP WR
REP	2	1 2
SAMPLE	3	1 2 3

Number of observations in data set = 12

NOTE: Observations with missing values will not be included in this analysis. Thus, only 8 observations can be used in this analysis.

The SAS System 7  
11:32 Tuesday, November 7, 1995

General Linear Models Procedure  
Repeated Measures Analysis of Variance  
Repeated Measures Level Information

Dependent Variable	C11	C25	C31	C39	C46
Level of DAY	1	2	3	4	5
Dependent Variable	C53	C60	C74	C81	C88
Level of DAY	6	7	8	9	10
Dependent Variable	C95	C109	C116	C129	C136
Level of DAY	11	12	13	14	15
Dependent Variable	C143	C150	C157	C164	C185
Level of DAY	16	17	18	19	20

The SAS System 8  
11:32 Tuesday, November 7, 1995

General Linear Models Procedure  
Repeated Measures Analysis of Variance  
Tests of Hypotheses for Between Subjects Effects

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TREAT	1	610.28926	610.28926	6.88	0.0394
Error	6	532.32566	88.72094		

The SAS System 9  
11:32 Tuesday, November 7, 1995

General Linear Models Procedure  
Repeated Measures Analysis of Variance  
Univariate Tests of Hypotheses for Within Subject Effects

Source: DAY

DF	Type III SS	Mean Square	F Value	Pr > F	Adj G - G	Pr > F H - F
19	1266.58029339	66.66212070	1.71	0.0448	0.2151	0.1699

Source: DAY\*TREAT

DF	Type III SS	Mean Square	F Value	Pr > F	Adj G - G	Pr > F H - F
19	1056.87795904	55.62515574	1.42	0.1294	0.2756	0.2487

Source: Error(DAY)

DF	Type III SS	Mean Square
114	4453.62357509	39.06687347

Greenhouse-Geisser Epsilon = 0.1241  
Huynh-Feldt Epsilon = 0.2437

The SAS System 10  
11:32 Tuesday, November 7, 1995

General Linear Models Procedure  
Repeated Measures Analysis of Variance  
Analysis of Variance of Contrast Variables

DAY.N represents the contrast between the nth level of DAY and the last

Contrast Variable: DAY.1

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	328.713774	328.713774	5.71	0.0541
TREAT	1	8.730140	8.730140	0.15	0.7105
Error	6	345.624149	57.604025		

Contrast Variable: DAY.2

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	134.407112	134.407112	21.56	0.0035
TREAT	1	2.320192	2.320192	0.37	0.5642
Error	6	37.404048	6.234008		

Contrast Variable: DAY.3

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	311.167923	311.167923	3.89	0.0960
TREAT	1	240.042893	240.042893	3.00	0.1339
Error	6	479.746587	79.957765		

Contrast Variable: DAY.4

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	278.705544	278.705544	1.78	0.2300
TREAT	1	226.280795	226.280795	1.45	0.2740
Error	6	936.926549	156.154425		

Contrast Variable: DAY.5

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	72.3023568	72.3023568	6.57	0.0427
TREAT	1	60.5460988	60.5460988	5.50	0.0574
Error	6	65.9951166	10.9991861		

Contrast Variable: DAY.6

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	80.1333691	80.1333691	1.53	0.2623
TREAT	1	29.8242953	29.8242953	0.57	0.4790
Error	6	314.1536336	52.3589389		

Contrast Variable: DAY.7

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	15.6175663	15.6175663	1.43	0.2763
TREAT	1	15.6175663	15.6175663	1.43	0.2763
Error	6	65.3441523	10.8906921		

Contrast Variable: DAY.8

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	1.08052350	1.08052350	4.24	0.0852
TREAT	1	1.08052350	1.08052350	4.24	0.0852
Error	6	1.52968521	0.25494753		

Contrast Variable: DAY.9

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	471.337412	471.337412	1.92	0.2153
TREAT	1	471.337412	471.337412	1.92	0.2153
Error	6	1473.641072	245.606845		

Contrast Variable: DAY.10

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	0.57171194	0.57171194	2.62	0.1565
TREAT	1	0.57171194	0.57171194	2.62	0.1565
Error	6	1.30810537	0.21801756		

Contrast Variable: DAY.11

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	0.79365511	0.79365511	2.45	0.1688
TREAT	1	0.79365511	0.79365511	2.45	0.1688
Error	6	1.94625740	0.32437623		

Contrast Variable: DAY.12

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	385.104652	385.104652	1.99	0.2078
TREAT	1	385.104652	385.104652	1.99	0.2078
Error	6	1159.989903	193.331650		

Contrast Variable: DAY.13

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	3.33178717	3.33178717	0.59	0.4708
TREAT	1	3.33178717	3.33178717	0.59	0.4708
Error	6	33.76400672	5.62733445		

Contrast Variable: DAY.14

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	0.06859993	0.06859993	0.11	0.7516
TREAT	1	0.06859993	0.06859993	0.11	0.7516
Error	6	3.74745618	0.62457603		

Contrast Variable: DAY.15

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	10.4598232	10.4598232	1.68	0.2427
TREAT	1	10.4598232	10.4598232	1.68	0.2427
Error	6	37.3845356	6.2307559		

Contrast Variable: DAY.16

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	1.45596874	1.45596874	0.44	0.5298
TREAT	1	1.45596874	1.45596874	0.44	0.5298
Error	6	19.65878147	3.27646358		

Contrast Variable: DAY.17

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	0.88851115	0.88851115	4.27	0.0843
TREAT	1	0.88851115	0.88851115	4.27	0.0843
Error	6	1.24843791	0.20807298		

Contrast Variable: DAY.18

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	0.14715041	0.14715041	0.45	0.5294
TREAT	1	0.14715041	0.14715041	0.45	0.5294
Error	6	1.98282010	0.33047002		

Contrast Variable: DAY.19

Source	DF	Type III SS	Mean Square	F Value	Pr > F
MEAN	1	0.49232468	0.49232468	2.98	0.1352
TREAT	1	0.49232468	0.49232468	2.98	0.1352
Error	6	0.99196629	0.16532771		

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